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Analysis of Mortars Used in Large-Scale Historic Masonry Walls: An Example from Northwestern Anatolia

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Abstract: The article examines the physical, petrographic, mineralogical, and microstructural properties of mortar samples taken from a medieval structure located in northwestern Anatolia. Six mortar samples collected from the structure were analyzed using advanced techniques such as acid loss, ignition loss, sieve analysis, physical analyses, polarizing and stereo microscope observations, SEM-EDS, XRD, and TGA. The mortars examined exhibit hydraulic properties. The hydraulic character of the mortars is mainly provided by brick dust and aggregates exhibiting pozzolanic activity. Acid loss and ignition loss analyses indicate that the binder-aggregate ratios vary between 1:1 and 1:3. The elemental and mineral composition of these mortars was analyzed using EDS and XRD, respectively. Analytical techniques revealed the presence of quartz, feldspar, muscovite, biotite, vaterite, and aragonite crystals. The results were supported by thermogravimetric analysis. This study provides important references for the formulation of compatible repair mortars to ensure the proper preservation of materials used in masonry walls of large-scale structures in similar geographical areas. It is intended that this study, based on the examination of mortar samples taken from the structure, will contribute to future research.

Key words: Masonry wall, Historical structure, building material, mortar, characterization.

1. Introduction

Monumental historical structures that represent the socio-economic conditions, cultural environment, and power of those who commissioned them during the period in which they were built constitute the most important and impressive examples in the history of architecture. These structures generally feature large masonry walls and attract attention with their durability and grandeur. The materials and construction techniques used in such structures play an important role in determining both the physical durability and aesthetic appearance of the structure. Today's restoration efforts highlight the need for a proper understanding of the material properties and technical applications of these structures, which have been built over the centuries, in light of historical information. As stated at the 2003 General Assembly of ICOMOS, it is necessary to fully understand and comprehend the characteristics of structures and materials in conservation practices. The properties of materials used in restoration (especially new materials) and their compatibility with existing ones must be thoroughly researched and understood, and the long-term effects of materials used in repairs must be investigated to prevent unwanted side effects. With this approach, this study examines the mortars used in the construction of the massive masonry walls of a medieval castle located in Balıkesir, which holds a unique position in northwestern Anatolia due to its geographical features and geological structure.

1.1 Historical Background

Large-scale walls and towers played a role in shaping cities in the early and medieval periods, and were particularly important elements of defense and military

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architecture in the Middle Ages [1, 2]. The masonry walls of these structures, constructed by stacking stones or bricks on top of each other and often joined together with mortar, have been preferred by various civilizations throughout history due to their durability and ease of construction. Numerous similar structures were also built in northwestern Anatolia during the same centuries. With its geographical features and geological structure, Balıkesir occupies a privileged position in northwestern Anatolia. It is located at the intersection of the north-south road connecting the Aegean and Marmara seas and the east-west road network [3-6]. One of the medieval castles built to secure these roads, which were used for military and commercial purposes, and to protect settlements is located in the Kadıköy neighborhood of Balya district. Researchers note that the structure was also used during the Hellenistic and Roman periods, but that it is mostly associated with Byzantine remains [3, 4]. The structure is fortified with double rows of walls on the southwest and west sides. The walls extending from west to north are built in a broken line with multiple corners due to the topography of the hill (Fig 1).

The walls were constructed using a double-layered stone masonry filled with rubble stone and mortar. In this type of wall, the wall has two surfaces, inner and outer, with the inner surface filled with rubble mortar; the inner and outer surfaces are made of cut stone or rough-hewn stone; the spaces between the surfaces (walls) are filled with rubble mortar [7]. The strength of the wall is ensured by the filling, which consists of a large amount of rubble stone mixed with lime-based mortar. The south-southwest towers and inner walls of the castle, which have survived to the present day while retaining their original layout, have largely preserved their outer walls. Upon examination of these sections, it was observed that three types of wall masonry were used in the structure. The two towers located in the south and southwest of the castle and the wall between them feature “ribbed masonry,” which is known to have become widespread in Anatolia during the Hellenistic

Fig. 1 General view of the structure.

Fig. 2 Types of masonry walls.

period [8]. Vitruvius defines this type of masonry as Greek-style masonry, or “emplekton” [9]. The “polygonal masonry” seen in the northern section of the inner wall line turns into “opus quadratum” towards the west. The “irregular masonry” found especially in the northern section is the third most common type of masonry (Fig 2). With the widespread use of lime mortar, which is a mixture of roughly slaked lime, sand, and water, the use of regular blocks in walls became unnecessary, and it was understood that walls could be easily built with irregular stones. The irregular wall masonry formed by stacking multi-sided stones with mortar is called “Opus incertum” [10, 11].

2. Method and Materials

2.1 Sampling

A total of six mortar samples were collected from points displaying different wall structures of the structure.Mortar samples were taken from the building's north, southwest, and west facades. The location where the mortar samples were taken on the building is shown in Fig 3.

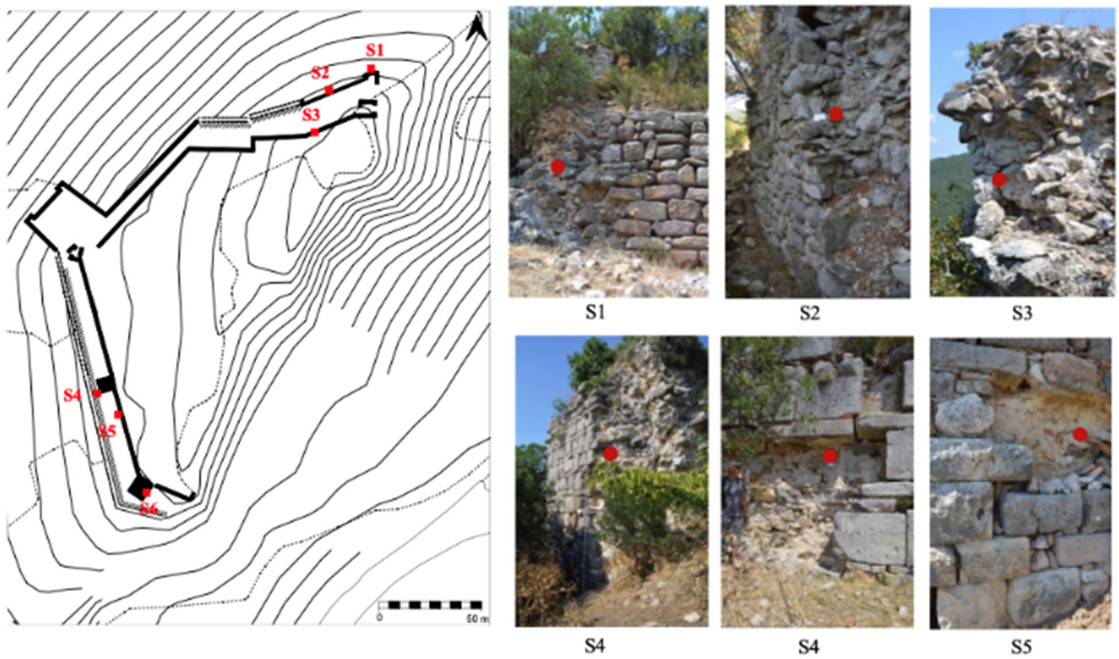

Fig. 3 Locations where mortar samples were taken from the structure.

2.2 Experimental Works

Number The experimental program included investigations of binder-aggregate ratio, aggregate size distribution, hydraulic properties of binders, the mineralogical and chemical composition of aggregates and binders and microstructural properties of mortars.

After the acid-loss, ignition-loss and sieve analysis of the collected samples, the silicate-based aggregate investigations were completed using a stereo microscope. The petrographic properties of the mortars were examined by polarized light microscopy and mineralogical analysis was performed by XRD. As a result of the physical tests, the actual and apparent density, porosity, and total water absorption values by weight and volume were determined. Hydraulicity was investigated by TGA analysis and finally, microstructure and elemental analysis were performed by SEM-EDS.

The basic physical properties of mortar samples were determined using standard test methods [12]. Real and apparent densities and porosity (total water absorption by volume) of mortars were determined according to EN 1936 standard and total water absorption by weight was determined according to TS EN 13755 standard [13-15]. A petrographic study was carried out using Polarized Light Microscopy to describe the samples' microstructural, compositional, and textural properties. There is no standard for this test; therefore, the recommendation of RILEM TC 167-COM (2001) was used [16]. For the analysis and observations, a stereo-microscope (single-nicol) was used to determine the macro-features, and a LEICA brand polarizing microscope (double-nicol) equipped with a digital camera was used to determine the micro-feature content of the thin section of the mortars.

The acid loss test was performed on approximately 20 g of mortar sample. Visual properties of aggregates such as colour, type and shape are determined using a stereo-microscope [17].

The hydraulic properties of the mortars were calculated from the weight loss percentages due to the molecular water (H2O) lost between 200 and 600 °C of the binders and the (CO2) released by the decomposition of the lime between 600 and 800 °C. For this purpose, powder samples prepared under <63 µm were measured by thermogravimetric analysis (TGA) using Perkin Elmer Pyris device. The analysis was carried out in a static nitrogen atmosphere between 25 and 820 °C at a heating rate of 10 °C/min. [18, 19].

X-ray diffraction (XRD) was used to characterize and semi-quantify the mineral phases detected in the different mortar samples used in the fortress. The Philips X'Pert Pro instrument determined crystal structure determination. Analyzes were performed on finely ground samples with a grain size of <125 µm.

The microstructural properties of the raw materials and binders exhibiting pozzolanic activity, the characteristics of the binder-pozzolan interfaces and the morphology of pozzolans, lime and binders were determined by scanning electron microscopy (SEM (Philips XL 30S FEG)) coupled with X-ray energy dispersive system (EDS). Before analysis, the samples were fixed to aluminium studs using carbon adhesive discs and coated with gold to ensure conductivity. Images of the samples were collected at different magnitudes (100x, 250x, 1000x, 2500x, 5000x) using a secondary electron detector at a voltage of 3 kV.

4. Results and Discussions

4.1 General Characteristics of Mortar

According to the physical analysis, the actual density of the mortars ranges between 1.3 and 2.0 g/cm3 and the open porosity between 31% and 43% (Table 1). These ratios are similar to those seen in mortar used in medieval structures [19, 20].

Raw material compositions of mortars were determined by binder/aggregate ratios and grain size distributions of aggregates (Table 2). The weight loss of the mortars at 105°C indicates the amount of water absorbed, varying between 0.55% and 7.21%. The weight loss fraction at 550°C, the ratio of molecular water and organic additive content (between 3.23% and 10.41%); The fraction lost at 1050°C indicates the CaCO3 ratio (between 26.85% and 68.01%). The values obtained are for lime (binder) and calcitic aggregates. According to the amount of CaCO3 provided by the acid loss analysis, the binder ratio of the mortars was determined as 30-60% by weight. Generally, all mortars' binder/ aggregate ratios vary between 1:1 and 1:3 by weight. Similar studies at the Byzantine structures of Rhodes, Venice, and Crete show the ratios of binder/aggregate (brick particles and dust) between 1:4 and 1:2 [21]. Ahunbay et al. (2003) [22] determined the binder-aggregate ratio as 1:2 for the 5th-century mortars from the Istanbul Walls and 1:3 for the 15th-century mortars; Güleç et al. (2013) [23] determined these ratios as 1:2 to 1:3 in their research conducted in another part of the same structure. In another study conducted in Yoros Castle (Istanbul), dating to the 13th and 14th centuries, ratios of 1:2 to 1:3 were observed [24]. As a matter of fact, it is seen that the binder/aggregate ratios in the mortars of historical buildings generally vary between 1:1 and 1:4 and even reach 1:5 in some Byzantine mortars with pozzolanic additives [19].

The mortar samples comprise grey-beige tones, medium-hard durable, heterogeneous size-distributed aggregates. Aggregates are mostly plagioclase-type feldspar, quartz, mica minerals and metamorphic and volcanic rock fragments. In addition, there are visible lime lumps in the mortar composition, where brick dust of different sizes is seen. (Fig 4).

Table 1 Basic physical properties of mortars.

Sample	Appearent density (g/cm^3)	Real density (gr/cm^3)	Open porosity (%)	Water absorption (weight) (%)	Water absorption (volume) (%)
1	2,26	1,33	41,15	29,96	39,89
2	2,27	1,57	30,72	18,93	29,79
3	2,25	1,35	39,90	29,37	39,79
4	2,24	1,34	40,26	27,76	37,07
5	2,22	1,53	31,29	20,32	30,99
6	2,39	1,37	42,51	22,59	31,04

Table 2 Basic Ignition loss and acid loss rates of mortars.

	Ignition loss			Acid loss		
Sample	105 C	550 C	1050 C	Loss (%) Binder	Remained (%) Aggregate	Binder: Aggregate
1	7,21	9,66	40,76	48,96	51,04	1:1
2	0,55	3,23	68,01	36,89	63,11	1:1,5
3	4,32	4,90	43,59	36,73	63,27	1:1,5
4	1,58	5,09	26,85	35,12	64,88	1:1,5
5	3,02	10,41	29,09	29,80	70,20	1:3
6	0,58	3,88	43,00	34,93	65,07	1:2

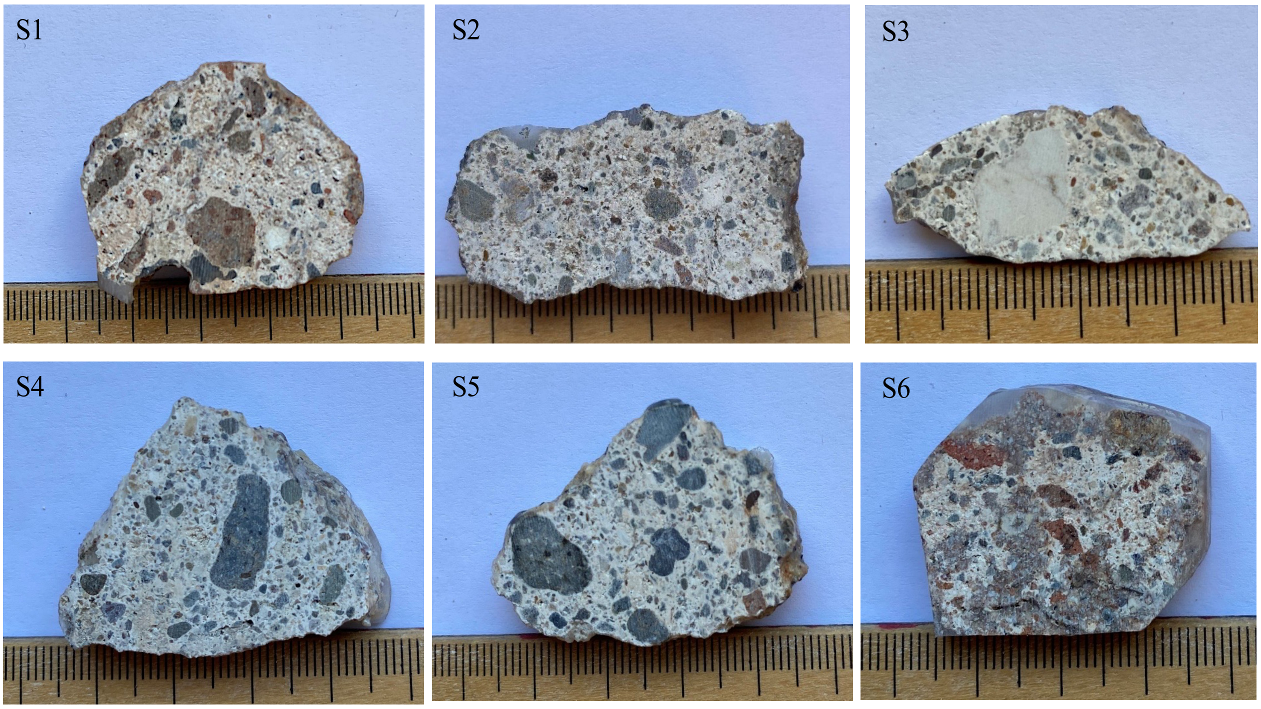

Fig. 4 Macro photography of mortars.

The siliceous aggregates of the mortars that did not react during the acid treatment were subjected to sieve analysis using the Turkish standard TS EN 1015-1 [25]. Aggregate-size distribution is given in Figure 5. They were examined under a stereo-microscope to determine their types and ratios. Generally, aggregates <125 µm consist of 5-10% brick dust, 10-20% biotite by weight and the rest quartz+feldspar. Aggregates between 500-1000 µm consist of a small amount of biotite, 20-25 wt% volcanic rock and 50 wt% quartz+feldspar. Except for coarse brick particles, 70-80% of the larger (>1000 µm) aggregates are gravel. Samples 2, 5 and 6 are below 8 mm sieve size. Samples 2, 3, 4 and 6 have similar aggregate size distribution. Sample 1 has a 500 µm sieve size distribution similar to these samples. Sample number 5 is quite different.

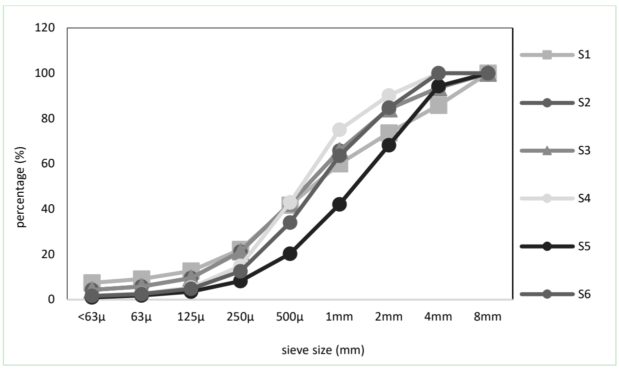

Fig. 5 Grain size distributions of the mortar aggregates.

4.2 Compositional and Textural Characterization

Mortar characterization was carried out by combining macroscopic observations and petrographic and micro-chemical techniques, as demonstrated in several studies focusing on this topic [26, 27].

4.2.1 Petrography

The petrographic properties of lime mortars may be according to factors such as quenching method, storage, lime lumps, shrinkage cracks, and carbonation rate [28, 29]. These properties of the lime in the mortar samples of structure were analyzed using polarized light microscopy on thin sections. Mortar samples consist of medium and well-sorted aggregates in a binder with a calcitic composition between 35-65%. The mortars are composed of a lime binder and aggregate of gravel-sized rock fragments, brick particles and powder, and siliceous sand. All mortar samples contain plagioclase-type feldspar and quartz minerals.

The binder of samples 2 and 6 is finer-grained than the others mortar compositions consisting of mineral, rock and brick fracture. Type of biotite and muscovite mica minerals are observed in samples 4-5-6; opaque minerals are observed in samples 1-2-3. The gravel-sized aggregates of the mortar samples consist of volcanic rocks such as basalt and andesite and metamorphic rocks such as gneiss and schist. Lime lumps are observed in samples 1-3 (table 3). The irregular structure of quartz and feldspar minerals suggests that the sand was transported by rivers.

Historic lime mortars are often characteristic of their location, both geographically and chronologically. The results of these petrographic studies supported the macroscopic observations. It allowed a better characterization of the textural properties of the mortars. In order to determine the source of the aggregates in the mortar composition, a sand-gravel mixture was collected from the Kocaçay River that flows around the fortress. Petrographic analysis of this mixture revealed that it contains plagioclase, orthoclase, quartz, biotite and opaque minerals. The gravel-sized pieces were identified as granitic-gneissic volcanic rock, sandstone, quartzite and limestone. It can be concluded that these results are compatible with the rock lithology of the region [30, 31].

4.2.2 Mineralogical Analyses

XRD investigated the general mineralogical composition of the samples. Mineralogical analysis results are given in figure 6. The main mineralogical components identified by XRD are calcite from carbonated lime and quartz. Carbonate fragments (vaterite, aragonite, dolomite) were also observed as aggregate. Although feldspars are observed in large amounts in petrographic analysis, the presence of feldspar minerals in XRD analysis is low. This situation can be explained as follows. The feldspars in the mortar composition are >125 microns in size. In addition to these minerals, mica minerals of muscovite type and opaque minerals in amorphous form were identified.

Table. 3 Petrographic properties of mortar samples (Qtz: quartz, Fsp: feldspar, Bt: biotite, Mscv: muscovite, Op: opaque mineral, Plg: Plagiclase, Ortho: orthoclase).

Samples	Location	Binder	Mineral	Lithic/ceramic fragments/addictives, Lumps
S1	north outer tower	calcite	Fsp (plg, orth), Qrtz, Op	Rocks (volcanic, conglomeratic), Quartzite, Brick fracture, lumps
S2	north outer wall	calcite	Fsp, Qrtz, Op	Volcanic rocks, Sandstone, Quartzite, Gneiss, schist, Brick fracture
S3	north inner wall	calcite	Fsp (plg, orth), Qrtz, Op, Bt	Micritic limestone, andasite-basalt, lumps, brick fracture
S4	southwest inner tover	calcite	Fsp (plg), Qrtz, Bt	Volcanic rocks, Gneiss, Schist
S5	southwesh inner wall	calcite	Fsp (plg), Qrtz, Bt, Mscv	Volcanic rocks, Gneiss, Schist, brick fracture
S6	south inner tower	calcite	Fsp (plg), Qrtz, Mscv	Peldspar phenocryst, Quartzite

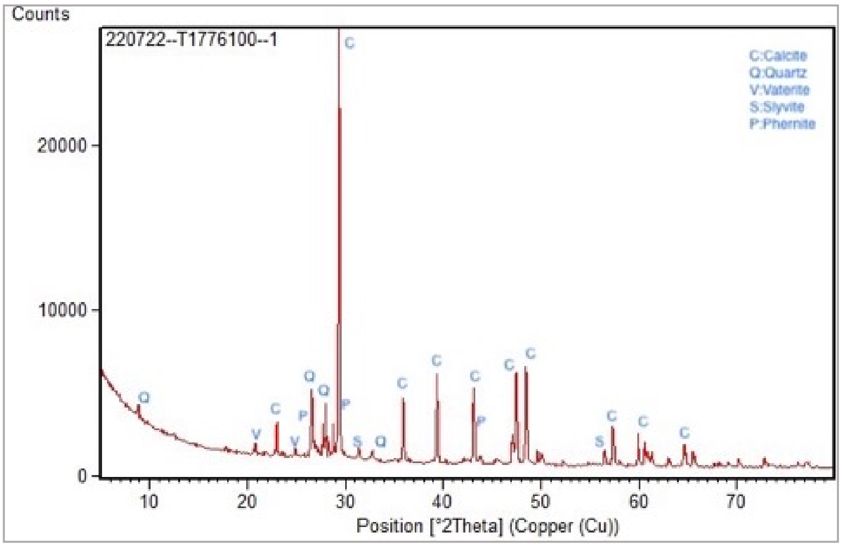

Fig. 6 Typical XRD model of samples (Sample 1).

Table 4 Elemental composition of binders in % oxides.

Sample	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	K_2O	MgO	Na_2O
1	28,89	47,68	9,78	2,34	3,23	4,63	1,38	1,24
2	63,72	23,37	6,95	1,60	-	1,69	2,22	0,45
3	34,26	47,94	10,20	4,78	-	1,47	1,00	0,33
4	22,00	47,70	14,29	9,30	-	4,30	0,29	2,13
5	51,99	36,04	6,54	1,21	-	0,60	0,79	0,97
6	22,91	43,13	11,08	4,39	13,97	3,18	1,04	0,29

4.2.3 Microstructural Analyses

The microstructure of the mortars, the reactions occurring at the binder/aggregate interface, and the bonds between the binder and pozzolan additives were examined with a scanning electron microscope (SEM).

The binders contain chemically large amounts of SiO2 and CaO; reasonable amounts of Al2O3; lesser amounts of Fe2O3, K2O MgO and Na2O (Table 5). While sample 4 contains a higher proportion of Fe2O3, SO3 was detected in the chemical composition of samples 1 and 6. This difference can be attributed to the chemical composition of the raw materials showing pozzolanic activity. However, it is not possible to make a precise inference with sufficient precision for statistical evaluation.

According to the EDX result, the binder contains both CaCO3 and siliceous material. This shows that the raw materials showing hydrated lime and pozzolanic activity result from silicification reactions. Sample 3 contains fibrous and needle-like CSH crystals (Figure 7). Dissolution and secondary interlayer pores were detected in the crystals. This may be mainly due to dispersed brick dust in the binder, which causes pozzolanic reactions that increase solubility [32, 33] (Fig 8). In addition, the analysis also revealed that the aggregates contain high levels of siliceous and aluminium, which is consistent with the data obtained in other analyses.

4.2.4 Thermogravimetric Analyses (TGA) and Hydraulicity of Mortar

The hydraulic properties of binders can be determined by TGA [34-36]. The results of TGA are shown in table 6. The table presents the weight loss percentages of the mortars at selected temperature ranges.

The CO2 / (SBW) ratio between the percentage weight loss attributed to CO2 (>600°C) and the percentage weight loss attributed to hydraulic water (200-600°C)

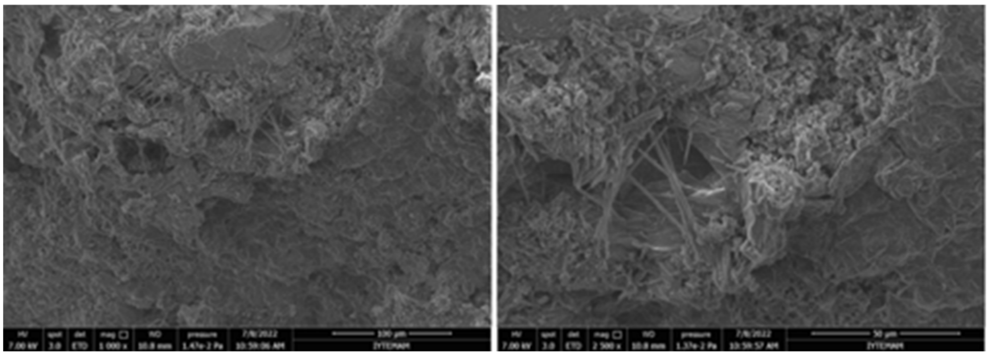

Fig. 7 Formation of fibrous and needle-like structure in sample 3.

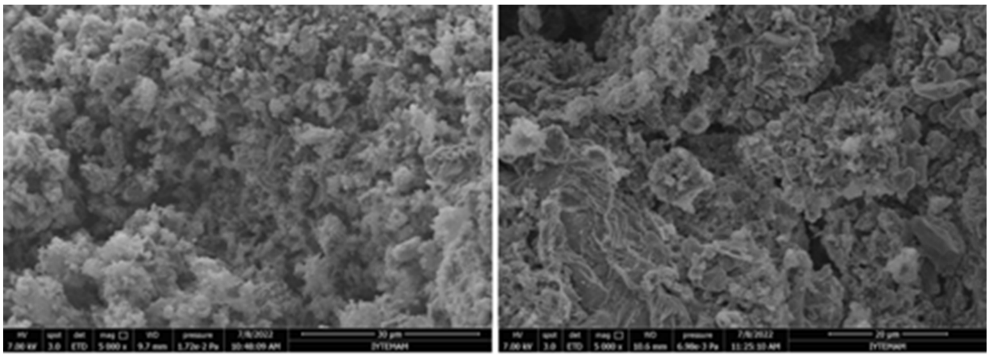

Fig. 8 SEM images of samples 1 and 4 showing the bond between calcite and pozzolan.

questions the hydraulicity of the mortar. When the weight losses due to CO_2 and SBW take a value between 1 and 10, it means that the mortar shows hydraulic properties [18, 37]. In the binary diagram of CO_2/SBW versus CO_2 of a mortar sample, if the CO_2/SBW ratio is less than five and the CO_2 is less than 15%, the mortar can be classified as high hydraulic. If the ranges are 15-25% for CO_2 and 5-10% for CO_2/SBW ratio, the compounds can be classified as hydraulic or artificial pozzolanic mortars [37].

Hydraulicity is one of the most important factors determining old mortars' durability and mechanical strength [18, 20, 33]. The hydraulicity of the mortar is due to the pozzolan added to the lime. The characterization of pozzolanic historic mortars has revealed that two types of pozzolans were used in the past. Natural pozzolans, usually of volcanic origin, and artificial pozzolans such as terracotta-ceramic powder [18, 21, 38].

The hydraulic properties of the mortars were also analyzed according to their microstructural properties. The products of hydraulic reactions (CSH and CAH) between pozzolans and lime were observed at the edges passing through pozzolans with widths <63 μm (e.g. Figure 9). EDS analysis revealed that these formations were mainly composed of CaO (22.0-63.72%), SiO_2 (23.37-47.94%) and Al_2O_3 (6.54-14.29%) (e.g. table 5). In addition, the factors affecting the pozzolanic activity of volcanic rocks are related to their content (SiO_2 + Al_2O_3 + Fe_2O_3), degree of crystallinity and fineness of their particles (Yu et al. 2015). Since most of the crystalline minerals of the volcanic rocks in the composition of the mortars are quartz and feldspar, it can be said that the pozzolanic activity component is SiO_2 and Al_2O_3.

The thermal analysis results show that all mortars are hydraulic when CO_2 /SBW ratios are calculated. When the CO_2 /SBW - CO_2 binary diagram is analyzed, it can be concluded that sample number 5 shows high hydraulic properties.

Table 5 TGA data for mortar samples (weight loss, %).

	Weight loss per temperature range (°C)					
Sample	<120	120-200	200-400	400-600 (SBW)	>600(CO2)	CO2/SBW
1	1,35	1,00	0,63	3,63	22,50	5,29
2	0,98	0,89	2,05	3,13	28,43	5,48
3	1,12	1,17	0,72	2,00	23,17	8,52
4	1,85	1,12	0,59	3,17	20,10	5,35
5	1,91	1,38	1,24	4,94	16,49	2,67
6	1,72	1,37	3,45	0,23	25,73	6,99

5. Conclusions

In this study, the physical, chemical, mineralogical, and microstructural properties of the original mortars used in the masonry walls of a large medieval structure in northwestern Anatolia were determined through various tests and advanced analyses.

Mortar samples taken from the fortress; have a solid structure and a heterogeneous texture. As a result of acid loss and heat loss tests, it was determined that the binder/aggregate ratios of the mortars were between 1:1 and 1:3; as a result of petrographic examinations and XRD analyses, the presence of quartz, cristobalite and muscovite minerals was determined in the mortars. As the binder, lime; as aggregate, it was determined to contain andesitic-basaltic rock and metamorphic rocks such as gneiss, schist, and brick fragments. In addition, the minerals determined due to the petrographic analysis of the aggregate samples taken from the river surrounding the building are similar to the minerals in the mortars' composition. This supported the idea that river sand and gravel may have been used as an aggregate.

According to the results of the TGA analysis, it was determined that the mortar samples examined showed hydraulic properties. While sample 5 shows good hydraulic properties, samples 1-2-4 have poor hydraulic properties. The ignition loss results also confirm this information. It is thought that the pozzolanic properties of the mortars may be due to the presence of quartz, cristobalite and muscovite minerals in samples 5 and 6 and brick fragments in the other samples.

In the SEM-EDS analysis, the presence of amorphous minerals and rod-shaped nanoparticles that increase the mortars' surface area confirms the mortars' hydraulic properties.

While collecting the mortar samples from the fortress, the sections with different masonry patterns were preferred as an approach to determine whether the building had different construction periods. According to the results of the analyses, the binder-aggregate ratio, aggregate-size distribution, hydraulicities and mineral structures of samples 2 and 3 taken from the north wall with polygonal masonry are similar. Samples 4-5-6 taken from the wall with opus incertum masonry have different characteristics from each other. In addition, samples 2 and samples 3 and 4 have similar compositions. These results suggest that the masonry and mortar compositions cannot be directly related. On the other hand, the difference in the masonry reinforces the idea that the building would have been rebuilt during the Byzantine period with the existing building materials as reported in the early researches on the building.

In conclusion, this study, which determines the original mortar properties of the structure and discusses the similarities and differences between mortar compositions, will be useful in preparing repair mortars to be used in conservation work on the structure. It also aims to contribute to the literature by revealing the original material properties of the period in which it was built.

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