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Investigating the historical building materials with spectroscopic and geophysical methods: A case study of Mardin Castle

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Abstract

Today, the building materials form the historical buildings are being exposed to various deteriorations increasingly due to different causes. Many historical masonry constructions in the world are on the edge of extinction due to the increasing frequency and changing models of material deterioration. The materials as close as possible to the original materials in terms of their chemical compositions and physical properties are required in the reconstruction and maintenance of the buildings that have historical importance. In addition, the properties of the materials used in the historical buildings are generally not known with a sufficient accuracy. This causes misapplications in case of emergencies, and also may lead to future potential greater damages on the building. The lack of data regarding the engineering properties of these buildings causes long-term damages on the buildings due to inappropriate conservation methods and materials. Therefore, it is necessary to investigate the properties of certain materials for application in the renewal of the historical buildings. Within this context, in this study the construction materials of Mardin Castle, which is located in Mardin Province, Turkey and existing for centuries as the symbol of the city, are investigated and its properties are reached. Experimental research methods were used in the study. Primarily, the castle structure was examined on-site by field study and sampling was carried out from the areas determined. The samples were analyzed via various spectroscopic and geophysical methods, and various findings were achieved. Relatively variable and high levels of salinization were determined. The findings regarding the average values in stone samples of Mardin Castle's Fortification Walls. Results of the research document the conservation status regarding Mardin Castle and provide an experimental base and also a theoretical support for the conservation of historical buildings in Turkey; and present indicative suggestions to establish conservation schemes of the historical buildings.

1. Introduction

The historical buildings are the most important elements that ensure the sustainability of the cultural heritage by witnessing the changes in culture and civilization [1-2]. However, today the building materials, which form the historical buildings, are being exposed to various deteriorations increasingly due to various causes. Stone building materials are affected by many deterioration mechanisms that are controlled by various factors such as the mineral composition, textural properties, pore / capillary structure, temperature, moisture, and exposure time with the environment that determines the complex physical, chemical and biological transformation processes [3-9]. A significant increase has been observed in abrasion rates of stone materials also with the air pollution increasing since the Industrial Revolution [10]. Today, damages caused on the cultural heritage are increased by increasing air pollution, human activities, and particularly by the release of the pollutants related with the industry, heating and traffic to the atmosphere [11-17]. Furthermore, also the great increase in CO_2 emission and the associated climate change in the last 150 years have negative impacts on the cultural heritage in various aspects [18]. It was seen that the global warming has increased the incidents causing the formation of harmful salines that affect the porous stone in the entire Central and North Europe [19-25]. Likewise, it was seen that advanced bioactivity of

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photosynthetic (micro) organisms (eg; cyanobacteria) with increasing CO_2 and the biological degradation, in which the most (micro) organisms are increased, are increased with the climate change [26]. For instance, colours of the stones have changed and foliation has been observed [27].

It is well known that also water plays a primary role in the deterioration processes of historical stones and mortars. Water causes disintegration, surface erosion, and cracking through the freezing – dissolution or wetting – drying circles within the pores by acting as a medium for the atmospheric pollutants such as sodium and nitrate that are increasing due to the reasons explained above. Furthermore, water may also carry the soluble salines, which trigger the crystallization between the pores and dissolution of the stones [9,28].

Increase of the material deteriorations on the stone materials and buildings by all these reasons has increased the need for the applications regarding the documentation and monitoring of material deterioration on the stone buildings. Today, unmanned aerial vehicle (UAV) and lidar technology can play important roles on documentation [29-31]. UAV and Lidar technology has been frequently used to document cultural heritage [32-34].

The resistance of the construction materials can be explained by the competent / incompetent status of the physical properties within the determined standards. Through the physical tests applied (such as hardness, unit volume weight, water absorption capacity, and porosity), it may be possible to determine whether the construction materials (particularly stone / rock) maintain their original qualities or to what extent they drifted away. Ultrasonic techniques are used in determining the dynamic properties of the rocks. These techniques are being used increasingly in construction technology due to the ease of application and non-destructiveness. The ultrasonic speed (SV) measurements, which are particularly being applied extensively on the rocks, provide important data also for the historical materials. The saline contents of the materials, which form the buildings, provide information that may be deemed as indicators on the physical status of the buildings. Salines, which are present within the content of different construction materials naturally or which are carried onto the surface or into the pores of the materials via the water by dissolving in water later as a result of capillary effect, provide information about the chemical changes that may occur both within the own structure of the material and within the structure of the other materials they interact.

In recent years, spectroscopy is also being used increasingly as an analytic technique for the researches on historical and cultural works to determine the stone building materials [35]. Spectroscopy is used to investigate the construction materials such as natural stones and mortars in architectural heritage and generally saline crystallization and black Shell formation, which are their deterioration types [36]. As an important example; in the study of Lodi et. al [37], it is aimed to obtain information about the composition of the mortar and stone materials, their conservation status and possible changing processes in situ on the samples received from a historical building in Venice via spectroscopy. It was concluded that the mortar and saline accumulations existing on the stone surfaces of the building have been crystallized very quickly due to the interaction of the abrasive atmospheric gases often with the nitrates and similar products.

Many historical masonry constructions in the world are on the edge of extinction due to the increasing frequency and changing models of material deterioration. The initial steps to set up an appropriate conservation treatment plan require the conservator to 1) determine the deterioration status through the microscopic and chemical analysis and 2) monitor the progress of deteriorations in time to determine the intensity and ratios [38].

The materials, which are as close as possible to the original materials in terms of their chemical compositions and physical properties, are needed in the reconstruction and maintenance of the buildings that have historical importance [39-40]. In addition, the properties of the materials [illegible] in the historical buildings are generally not known with a sufficient accuracy. This causes misapplications in case of emergencies, and also may lead to future potential greater damages on the building. The lack of data regarding the engineering properties of these buildings causes long-term damages on the buildings due to inappropriate conservation methods and materials [41]. Therefore, it is necessary to investigate the properties of certain materials for application in the renewal of the historical buildings. Within this context, in this study it is aimed to document the construction materials, to determine the material problems, and to present conservation suggestions through the studies carried out by spectroscopic and geophysical methods on the materials of Mardin Castle's Fortification Walls. The construction materials of Mardin Castle, which is located in Mardin Province, Turkey and existing for centuries as the symbol of the city, are investigated and its properties are reached as a result of the study. The findings of the study do not only reflect the conservation status of the historical building in real terms, but also provide an experimental base and theoretical support for the conservation of the historical buildings in Turkey. It presents indicative suggestions to establish conservation schemes of the historical buildings and enriches the maintenance and reinforcement evaluation status of the historical buildings.

Within this scope, initially information regarding the working area is presented in the section below. Then, the method followed in the study is described and the findings are presented. In the next stage, various results are achieved by comparing the findings of the study with the data obtained in the literature. In the conclusion section of the study, various conservation suggestions are presented regarding the building.

1.1. Location and importance of the study area

Mardin settlement, which has the characteristic of monumental city today, is consisted of two elements. These are Mardin Castle and the essential settlement developed on the plinth of the castle. The Castle is located on the thin-long plane on a hill having a height of 1200 metres. This plane is in a size of 800 m in east-west direction, and of 150 m in the widest place and of 30 m in the narrowest place in North –south direction. The Castle gives the impression of a natural formation, since it has been built so as to involve the existing rocks with its walls and towers. Entry is made to the castle, where the defence capacity was strengthened, from a point that nearly centres area, on which the castle is placed, from the South, to the extent allowed by the topography. This entrance is accessed through a ramp, which steepens gradually, and stairs located at the end of it [42] (Fig. 1).

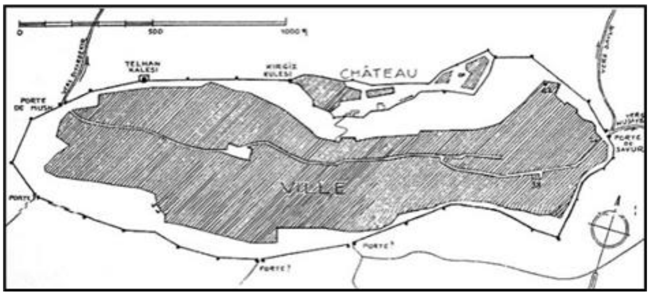

Figure 1. Plan of Mardin city walls charted by Gabriel [42]

Access to the main entrance door of the Castle from the historical settlement located on the South slope of Mardin Castle is made from a central point allowed by the topography. Firstly, the historian Ammianus Marcellinus has mentioned the Mardin Castle, which has gain reputation as a place hard to occupy along the history, the IV. Century AC. Mardin Castle has begun to be mentioned in the historical records after a long time by Arabic scholars only from the X. century. In this period, Mardin Castle has been called Şahin Castle or Karg[illegible] Castle. In 1471, it has been mentioned by the Merchant Barbaro, who has come to Mardin, in his memories that the castle had walls exceeding 12 m. and it was accessed by the stairs, and there had been nearly [illegible]00 houses within the interior castle. As of XVII. Century, according to Evliya Celebi, it has been reported that the Castle has gone under many repairs, and grain has been stored in its caves and cellars, and there had been water cisterns.

The Castle has been found by Niebuhr, who has come to Mardin in XVIII. Century, as undamaged enough, but neglected, according to the observations of Niebuhr, it has been estimated that nearly 200 houses were present, of which 80 could be habitable. In 1891, according to the records of Cuinet, it has been reported that there were 4991 houses in total within the castle and the city. Some monumental structures in Mardin Castle, in a ruin status, and the “mansion” structure as an example of the civil architecture have been emphasized in the studies conducted by Gabriel in the region in 1930. Today, the mansion structure, which has been documented by Gabriel, is destroyed. The visible relics belong to the Castle’s Mosque, which is placed just above the castle entrance and thought to be built in XV. Century, within the period of Akkoyunlular over the Artuqid building and to the Hızır Mosque, which is believed to belong to the period of Khalif Ömer.

The Mardin city, which has been developed on the plinth of the castle, is among the rare Anatolian cities that have conserved the traditional city texture. Today, the entire region, in which the Castle and the old neighbourhoods that the traditional city texture is conserved are placed, remains within the urban archaeological site. The greatest factor in the conservation of the traditional city texture is definitely the ethnical building features of the neighbourhoods, which have been conserved within the historical process (“Fig. 2”).

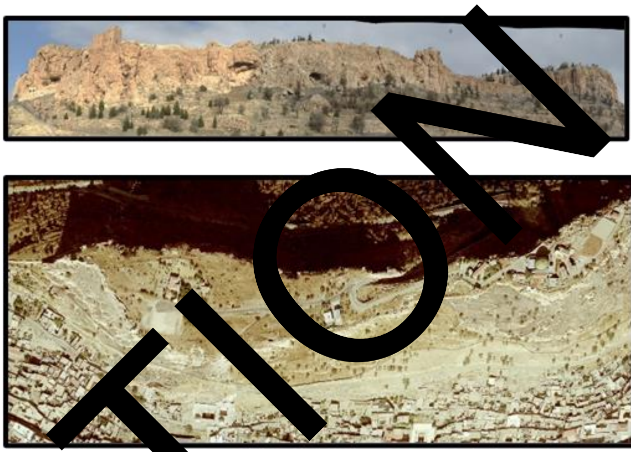

Figure 2. Mardin Castle

2. Method

Construction materials of Mardin Castle’s Fortification Walls were investigated with various methods within the scope of the research. Experimental research methods were used in the research. Primarily, the castle structure was examined on-site by field study and sampling was carried out from the areas determined. The samples were analyzed via various spectroscopic and geophysical methods, and various findings were achieved.

In the first stage of the research, physical tests, which aim to determine the unit volume weight and porosity properties, were applied on the stone/rock and ceramic (brick) samples. In the second stage, total saline content of the stone and brick samples of the fortification walls were determined conductometrically. Afterwards, the water-soluble saline types (phosphate and carbonate) and the environmental pH values of the samples (stone and brick) were determined quantitatively. It was found that the saline contents of the samples, which reflect the basic environmental conditions, demonstrated carbonation in high values, via the spot saline tests. Aggregate particle distribution values were obtained by the total aggregate and binding agent ratios and the aggregate granulometrics by acidic aggregate / binding agent analysis in the samples of mortar and plaster. Rock and mineral content, texture, status, distribution, particle sizes of the samples were investigated by petrographic fine section optical microscope analysis. Chemical content of all structural samples of the Castle were determined by PED-XRF analysis. Cementation Index data was used in order to determine the type of limestone in the mortars and plasters (“Fig. 3”).

Experiments, which were conducted under the research, are stated below, in summary.

- Documentation (Photographing), Coding and Cataloguing (Material Grouping) Studies
- Physical Tests (Unit Volume Weight, Water Absorption Capacity (WAC), Porosity, Schmidt Hardness, Ultrasonic Speed)
- Conductometrical Analysis (Amount of Total Water-Soluble Saline)
- Saline Type Tests (SO_4^{2-}, Cl^-, PO_4^{3-}, CO_3^{2-}, NO_3^- and NO_2^-)
- Aggregate / Binding Agent Tests
- Granulometric Analysis in Aggregates (Particle Size Distribution)
- Petrographic Fine Section Optical Microscope Analysis
- X-Ray Fluorescence Analysis (M-XRF)
- X- Ray Fluorescence Analysis (PED-XRF)

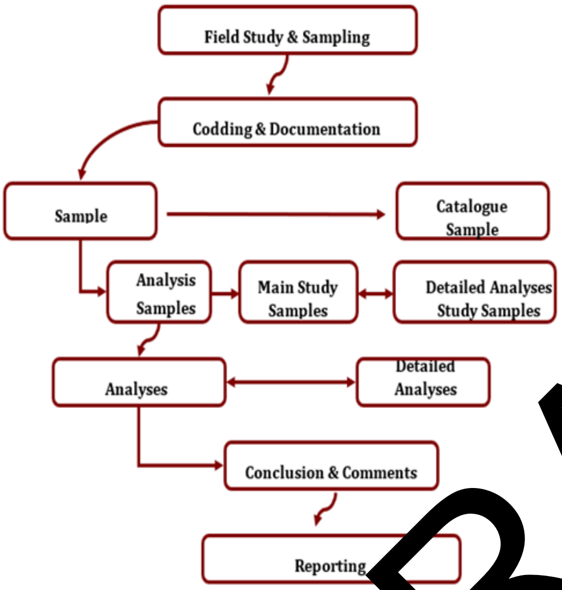

Figure 3. Material Investigation Methodology of Mardin Castle's Fortification Walls

2.1. Constructional material groups of Mardin Castle's fortification walls

Firstly, castle structure was investigated on site, and places determined on the structure were codded and sampling was carried out. Places, where the samples were taken on the Castle, are stated in "Fig. 4". Materials taken from the coded places were grouped. Material groups are [illegible] in "Fig. 5". Analyses applied on the material groups are described in "Table 1".

Table 1. Analyses applied on the material groups during the research

Material Group	Applied Analyses / Tests
Stone / Rock Samples	1, 2, 3, 4, 7, 9
Ceramic Samples	1, 2, 3, 4, 7, 9
Mortar Samples	1, 5, 6, 7, 9
Plaster / Plaster Layer Samples	1, 5, 6, 7, 9
Soil Sample	1, 3, 4, 9
Lime Layer Sample	1, 9

1. Documentation (Photographing), Codding and Cataloguing (Material Grouping) Studies
2. Physical Tests (Unit Volume Weight, Water Absorption Capacity, Porosity, Schmidt Hardness, Ultrasonic Speed)
3. Conductometrical Analysis (Total Amount of Water-Soluble Saline)
4. Saline Type Tests (SO42-, Cl-, PO43-, CO32-, NO3- and NO2-)
5. Aggregate / Binding Agent Analysis
6. Granulometrical Analysis in Aggregates (Particle Size Distribution)
7. Petrographic Fine Section Optical Microscope Analysis
8. X-Ray Fluorescence Analysis (M-XRF)
9. X- Ray Fluorescence Analysis (PED-XRF)

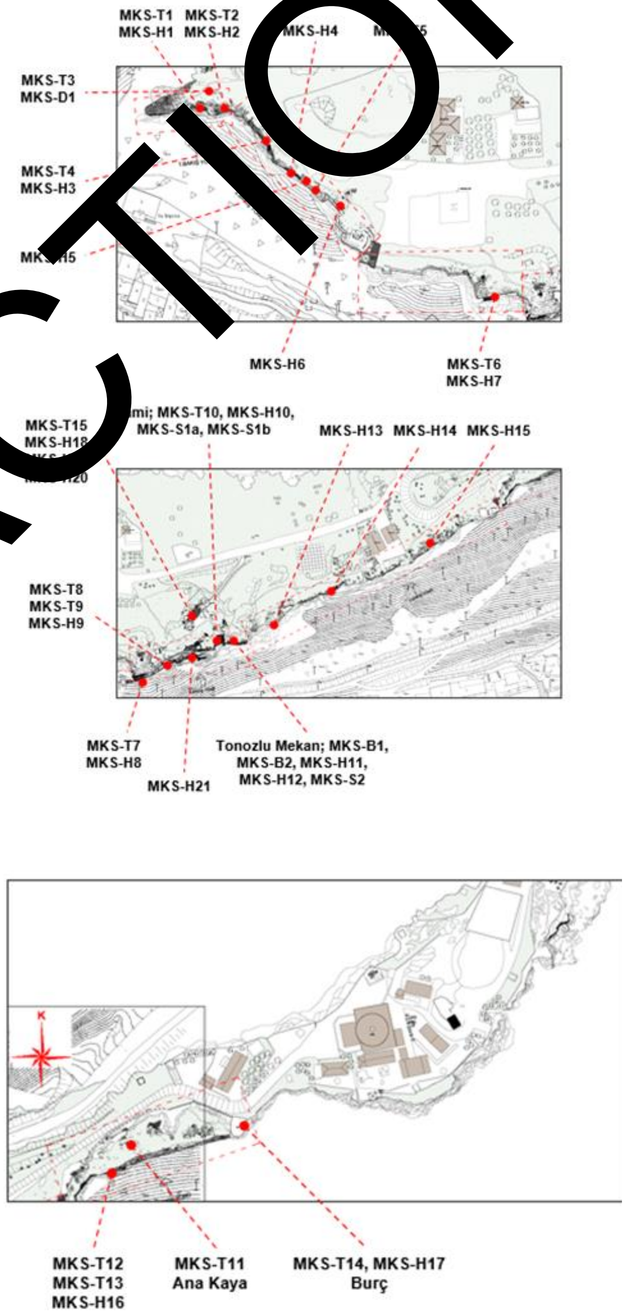

Figure 4. Coding of the Samples taken from the Castle on the layout plan

Material Group Code	Material Group Descriptions	Number of Main Samples
MKS-T	Stone / Rock Samples	25
MKS-B	Ceramic (Brick) Samples	2
MKS-H	Mortar Samples (From Stone/Brick Pointing and Debris Fillings)	31
MKS-S	Plaster/Plaster Layer Samples	3
MKS-D	Soil Sample	1
MKS-Z	Lime Layer Sample	1


Stone Sample


Brick Sample


Mortar Sample


Limestone Layer


Plaster Sample

Figure 5. Constructional Material Groups of Mardin Castle's Fortification Walls

3. Results

3.1. Physical Tests

3.1.1. Unit Volume Weight, WAC and Porosity

Physical statuses of the materials were determined through the physical tests applied on the stone/rock and brick samples of Mardin Castle's Fortification Walls. Since the sample amount required to conduct the standard physical tests (standard samples of 5-10 cm^3; RILEM, 1980) was not possible in terms of the standard applications, the test applications were conducted on the stone pieces taken by sampling. Basic physical tests, which aim to determine the unit volume weight, water absorption capacity and porosity, and hardness (Schmidt and ultrasonic speed (SV) measurement tests were applied on the stone samples. For the basic physical tests, unit volume weights (wet/dry UVW, g/cm^3), water absorption capacities (%WAC) and porosity (%P) values were determined by means of the dry weights taken directly, archimedes (within water) and saturated weights (watery weight, which it is ensured to reach to the pores under 50 torr pressure in distilled water) of the samples ("Table 2" and "Fig. 6-7"). Stone samples have physical properties changing depending on the natural rock structures, and all samples have physical properties changing depending on the environmental properties. The samples having low unit volume weights and high porosities with their structural properties are those in a more incompetent states. The stone samples of the fortification walls are among the limestone rock types. Among the stones, MKS-T5, MKS-T[illegible] and MKS-T10 (Biosparitic Limestone) samples are those having the lowest competency and MKS-T9 (Argillaceous Limestone) is the one having the highest competency (Table 2).

Table 2. Unit volume weights (wet/dry UVW, g/cm^3), water absorption capacity (%WAC) and porosity (%P) values

Samples	UVW-I (g/cm^3)	UVW-K (g/cm^3)	WAC (%)	P (%)	Type
MKS-T1	2.60	2.35	3.96	9.33	R. Limestone
MKS-T2	2.73	2.34	6.07	14.20	A. Limestone
MKS-T3	2.61	2.20	7.18	15.79	A. Limestone
MKS-T4	2.75	2.35	6.16	14.46	A. Limestone
MKS-T5	2.43	1.95	10.20	19.89	B. Limestone
MKS-T6	2.50	1.93	11.81	22.80	B. Limestone
MKS-T7	2.66	2.40	4.01	9.63	Travertine
MKS-T8	2.69	2.31	6.10	14.11	A. Limestone
MKS-T9	2.72	2.63	1.26	3.32	A. Limestone
MKS-T10	2.51	1.99	10.57	21.00	B. Limestone
MKS-T11	2.68	2.40	4.34	10.43	A. Limestone
MKS-T12	2.70	2.53	2.50	6.32	A. Limestone
MKS-T13	2.66	2.47	2.90	7.18	A. Limestone
MKS-T15	2.55	2.49	1.00	2.50	Travertine
MKS-T16	2.52	2.34	3.15	7.37	R. Limestone
MKS-T17	2.49	2.13	6.78	14.43	R. Limestone
MKS-T18	2.54	2.28	4.46	10.18	A. Limestone
MKS-T19	2.62	2.31	4.96	11.48	A. Limestone
MKS-T20	2.66	2.54	1.77	4.49	A. Limestone
MKS-T21	2.59	2.45	2.21	5.40	A. Limestone
MKS-T22	2.30	1.99	6.92	13.76	A. Limestone
MKS-T23	2.57	2.33	4.07	9.48	A. Limestone
MKS-T24	2.52	2.02	9.80	19.83	B. Limestone
MKS-T25	2.67	2.51	2.42	6.08	A. Limestone
MKS-B1	2.30	1.39	28.19	39.30	Brick
MKS-B2	2.26	1.38	27.92	38.65	Brick

B. Limestone: Biosparitic Limestone,
A. Limestone: Argillaceous Limestone,
R. Limestone: Recrystallized Limestone

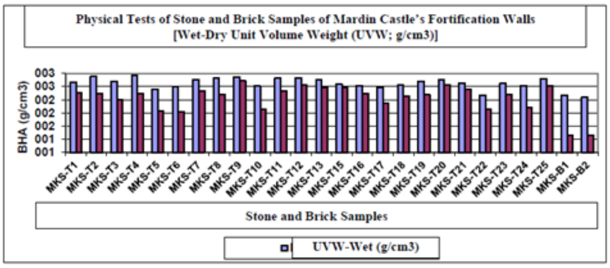

Figure 6. Unit volume weights (wet/dry UVW, g/cm^3)

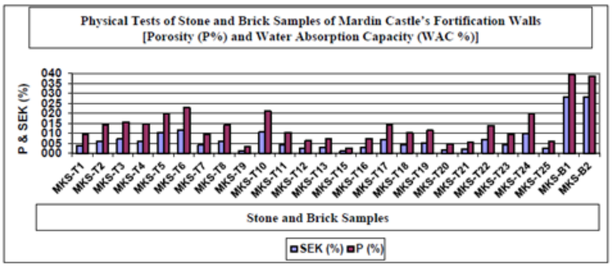

Figure 7. Water absorption capacity (%WAC) and porosity (%P) values

3.1.2. Hardness Test

Schmidt Hammer is used to determine the hardness value of the rocks. The hardness value determined is used in UCS estimation and classification of the rocks. However, this method cannot be applied on very soft or very hard rocks. During the test, attention was paid to maintain the hammer always in a vertical position [illegible] the rock. In the test, strokes were made on the 5 points on the rock surface, and average hardness values were obtained. Digital Proseq brand Schmidt Hammer was used in the measurement. Schmidt Hammer measurement results of the stone samples (limestone) of Mardin Castle's Fortification Walls support[illegible] basic physical tests. Stone hardness of [illegible]3 sample is higher than the other samples (26.3), and hardness of MKS-T1 stone sample is lower (22.0). If we evaluate in general, stone hardness of the samples are very close to each other ("Table 3" and "Fig. 8").

Table 3. Stone hardness measurement tests

Samples	M 1	M [illegible]	M 3	M 4	M 5	Average
MKS-[illegible]	[illegible]	[illegible]	[illegible]	22.3	22.5	22.0
MKS-T2	[illegible]	24.6	25.8	26.3	26.8	25.3
MKS-T3	[illegible]	[illegible]	26.2	26.3	26.5	26.1
MKS-[illegible]	24.0	24.2	25.0	25.2	25.3	24.7
MKS-T8	25.3	25.3	25.4	25.5	25.7	25.4
MKS-T20	22.0	23.1	23.7	24.0	24.5	23.5

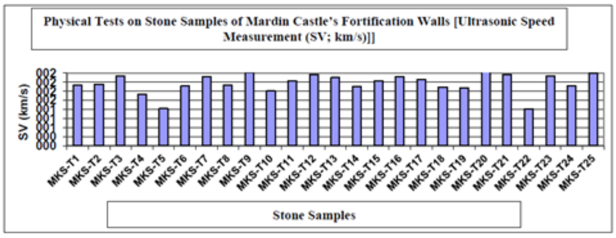

Figure 8. Hardness Test

3.1.3. Ultrasonic Speed Measurement Test

Measurements were taken by using Matest C372N Model High-Performance Ultrasonic Test Device. SV measurements conducted on the stone samples, taken from Mardin Castle's Fortification Walls are presented in Table 4c over the average values. It could be determined in the measurements that some of the limestone samples were more porous and in the stage of deterioration. Among the samples, those having higher SV (km/s) values are more competent. This is in compliance with the data, which reflects the basic physical properties of the same samples. Within the sample set, MKS-T22 sample has the lowest competency [illegible] sample has a higher competency ("Table 4" and "Fig. 9").

Table 4. SV measurements of the limestone samples

Samples	SV (µs)	SV (km/s)	Samples	SV (µs)	SV (km/s)
MKS-T1	41.6	2.00	MKS-T14	42.2	1.96
MKS-T2	[illegible]	[illegible]	[illegible]	39.7	2.15
MKS-T3	38.0	[illegible]	MKS-T16	57.0	2.28
MKS-[illegible]	[illegible]	1.71	MKS-T17	39.3	2.19
MKS-T5	[illegible]	1.23	MKS-T18	47.6	1.94
MKS-T6	42.1	1.97	MKS-T19	46.5	1.90
MKS-T7	38.2	2.29	MKS-T20	54.2	2.44
MKS-T8	41.7	2.00	MKS-T21	41.0	2.35
MKS-T9	36.9	2.42	MKS-T22	59.1	1.22
[illegible]	44.5	1.81	MKS-T23	38.0	2.31
MKS-T11	39.7	2.15	MKS-T24	32.6	1.99
MKS-T12	39.6	2.36	MKS-T25	40.4	2.40
MKS-T13	38.6	2.25	Average	**43.4**	**2.07**

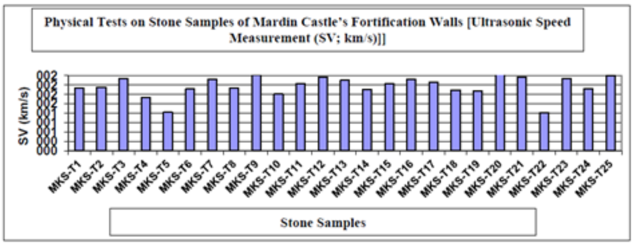

Figure 9. Ultrasonic speed measurement test values

3.2. Conduktometrical Analysis (Total Amount of Water-Soluble Saline)

The total amount of water-soluble saline present in the structure (pores) of the stone and ceramic (brick pieces) samples of Mardin Castle's Fortification Walls was determined quantitatively ("Table 5" and "Fig. 10"). For the designation of total saline measurement in the samples; sample of 5 grams, which was taken into 25 ml of water, was centrifuged for 1 hour and standard sodium hexametaphosphate was added over it after filtering. Total saline contents of the samples prepared were recorded conductrometrically via the conductometer (with Neukum Series 3001 brand conductivity/pH/heat meter). In the widest meaning, the saline content within the materials is caused by the environment of the samples, by the petrographic rock properties for the stones / rocks or natural or metabolous (deteriorated) physical / chemical properties of the materials. The total saline content of stone /rock samples of Mardin Castle's Fortification Walls varies between 0.48-3.22% (average 1.53) and is between 0.27% and 1.22% for brick samples ("Table 5" and "Fig. 10"). Excessively variable and high levels of salination were determined in stone samples in terms of average values. Among the stone sample set, low salination was determined in MKS-T10 (limestone) sample (0.48%), and very high salination was determined in MKS-T22 (limestone) sample (3.22%) ("Table 5"). For MKS-T22 sample, this refers to a very advanced level of salination.

Table 5. Total Amount of Water-Soluble Saline

Samples	SS (%)		
MKS-T1	0.65	MKS-T15	2.26
MKS-T2	0.83	MKS-T16	2.01
MKS-T3	1.04	MKS-T17	2.29
MKS-T4	0.86	MKS-T18	1.66
MKS-T5	2.04	MKS-T19	1.10
MKS-T6	2.85	MKS-T20	0.95
MKS-T7	1.98	MKS-T21	0.84
MKS-T8	1.40	MKS-T22	3.22
MKS-T9	1.50	MKS-T23	1.73
MKS-T10	0.48	MKS-T24	0.97
MKS-T11	0.99	MKS-T25	0.62
MKS-T12	2.10	MKS-B1	0.2[illegible]
MKS-T13	1.17	MKS-B2	1.[illegible]
MKS-T14	2.82	Stone Average	1.53

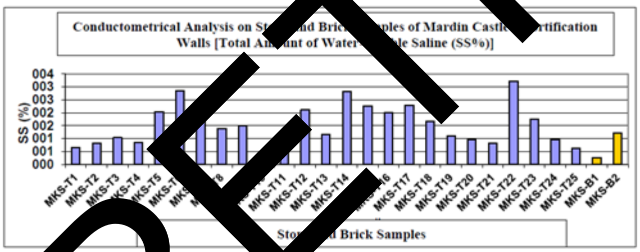

Figure 10. Total Amount of Water-Soluble Saline

All samples of Mardin Castle's Fortification Walls are open to the impact of high salination. The impact of the climatic circle is important at this point (Table 6). When long-term weather conditions of March in Mardin is considered, on average 10.7 days per month is rainy (Table 6). The heavy rains between December and April in Mardin are relatively higher than the summer season. Average temperature is 8.3°C in March and hours of sunshine are 6.1 hours during the day (Table 6). Formation of relatively humid environmental conditions were observed in the stone samples, which have been exposed to water accumulation with the heavy side rains, during the sampling carried out in the last period of the winter season, which was rainy and in which the sun showed its face less. This causes an impact, which accelerates the deterioration on the stones. The humid environment formed on the stones causes the limestone stratification, which occurs with the recrystallization of the salines dissolved by lichenification /vegetation on the stone surfaces, with the impact of microclimate, and high salination associated with this, and reveal of destructive effect of salination on the stones (dissolution, disintegration).

3.3. Analysis of Saline Type (Water-Soluble Saline Types in Stones and Ceramics)

Types and amounts of the salines determined by the standard (Merck) spot saline type tests are presented in Table 7, and environmental pH values are presented in Table 7 and Graphic 7.

pH Distribution in the Samples: It was determined that the pH values of the stone/rock samples investigated (independent from the rock type) vary between 7.03 – 8.66, pH values of brick samples are between 7.81 and 7.95, of soil sample is 8.[illegible]3 and of limestone layer sample is 6.64. The samples investigated have a poor basic feature in general, on the other hand the limestone layer sample has a more acidic feature than the other samples (Table 7 and Graphic 7).

Carbonate Test (CO32-): It helps to determine the binding agent containing lime in mortar and plasters. It is used in determining the combination of stones containing carbonate (marble, travertine, limestone, etc.) and calcified surfaces in stones/rocks. Carbonation varying between 11.2 - 80 mg/L was found in the samples. The high carbonate content determined in the soil sample, which represents the reservoir (80 mg/L), is a criterion for the other materials (Table 7).

Phosphate Test (PO43-): The impact of agricultural activities (fertilization containing phosphate), animal (defecation) or vegetable residues, sewage or household wastes may be caused by transportation of food deposits, directly or indirectly, from the soil reservoir to the material by moisture, in the vicinity of the wastes or picnic sites. Furthermore, high phosphate content is observed in rock structures or mortars and plasters with organic (plant – straw) content, and also in the materials that are exposed to the effects of lichenification on the surfaces with intense humidity.

Although it is determined in relatively low amounts in only 2 of the stone samples of the Castle (0.20), higher amounts of phosphate were determined in brick, soil, and limestone layer samples ("Table 6" and "Fig. 11").

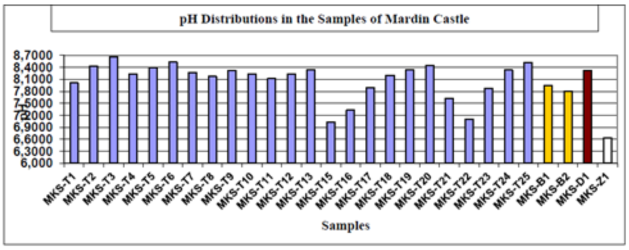

Figure 11. Saline Type Analysis (Water-Soluble Saline Types in Stone and Ceramics)

Table 6. Values of Saline Type Analysis

	Phosphate (PO_4^{3-})	Carbonate (CO_3^{2-})	pH
MKS-T1	-*	11.2*	8.01**
MKS-T2	-	40	8.44
MKS-T3	-	80	8.66
MKS-T4	-	19.2	8.23
MKS-T5	-	19.2	8.40
MKS-T6	-	40	8.53
MKS-T7	-	59.2	8.26
MKS-T8	-	19.2	8.17
MKS-T9	-	40	8.32
MKS-T10	-	59.2	8.23
MKS-T11	0.20	19.2	8.12
MKS-T12	-	40	8.24
MKS-T13	-	40	8.34
MKS-T15	-	19.2	7.03
MKS-T16	-	19.2	7.33
MKS-T17	-	19.2	7.90
MKS-T18	-	59.2	8.20
MKS-T19	-	11.2	8.35
MKS-T20	-	59.2	8.45
MKS-T21	-	19.2	[illegible]
MKS-T22	-	11.2	7.[illegible]
MKS-T23	0.20	11.2	7.88
MKS-T24	-	59.2	8.33
MKS-T25	-	80	8.52
MKS-B1	0.20	59.2	7.95
MKS-B2	0.40	40	7.81
MKS-D1	0.80	8[illegible]	8.33
MKS-Z1	0.20	19.2	6.64

3.4. Aggregate / Binding Agent and Granulometrical Analyses

Samples, which were subjected to dry weighing initially in order to determine the aggregate and binding agent parts of 20 mortar and 2 plaster layers samples taken from Mardin Castle's Fortification Walls, were treated with diluted acid (HCI of 5%) in order to refine them from binding agent contents (all carbonate content; CO32-) later (1).

CO32- (aq) + 2HCl (aq) →CO2 (g) + Cl- (aq) + H2O (s)

After the mortar and plaster layer samples, which were separated from lime and all carbonate contents (binding agent) by the filtering, washing, and drying procedures) and where the aggregate part was obtained, are dried at room temperature, and total binding agent and aggregate amounts in terms of weight were obtained by subjecting to re-weighing ("Fig. 12"). Aggregate particle distributions were determined (granulometrical analysis) by applying systematic sieving to the aggregates of the samples (which do not contain carbonate) ("Fig. 13"). All mortar and plaster samples, which allowed this analysis, were subjected to the abovementioned analyses (TS 3530 –Particle Size Distribution Designation – Sieving Method).

Supportive fine section optical microscope analyses and acidic aggregate / binding agent analyses were detailed, and amounts, types and distribution of aggregate and binding agent were obtained in mortars and plasters, and the samples were grouped (Table 7, Fig. 12-13).

When the aggregate content of 20 mortar and 2 plaster layers samples of Mardin Castle's Fortification Walls, independent from being original or repaired, are reviewed in detail, it was found that the aggregate structure of majority of the analysed mortars were consisted of coarse particles (500-1000 µm; containing coarse sand and small stone pieces). Besides, an aggregate mixture formed by [illegible] aggregates collectively, which demonstrates an average and more balanced distributions (63-500 µm), were also present in the pointing stuff and mortar samples.

Table 7. Aggregate / Binding Agent and Granulometrical Analyses

Samples	<63 µm	>63 µm	>125 µm	>250 µm	>500 µm	>1000 µm
MKS-H1	13.47	9.07	14.02	22.18	31.39	9.86
MKS-H3	12.35	9.55	15.67	24.36	35.41	2.66
MKS-H4	11.03	8.44	13.71	24.05	37.07	5.69
MKS-H5	10.65	9.31	15.24	24.53	33.05	7.23
MKS-H8	13.60	9.38	11.64	18.85	32.95	13.58
MKS-H9	14.76	9.22	13.30	20.62	34.73	7.37
MKS-H10	19.10	10.51	13.52	19.65	24.84	12.38
MKS-H11	15.64	6.92	10.50	17.65	36.10	13.18
MKS-H12	15.02	9.60	16.84	25.98	26.33	6.22
MKS-H14	12.34	4.97	8.57	14.86	20.77	38.48
MKS-H15	10.86	7.23	12.13	22.17	30.65	16.96
MKS-H16	15.80	11.29	15.39	20.87	25.89	10.76
MKS-H18	11.56	8.10	13.35	21.50	29.38	16.11
MKS-H21	15.98	7.12	10.61	17.24	29.57	19.49
MKS-H24	14.44	8.59	12.53	17.76	29.09	17.60
MKS-H26	13.97	8.18	13.47	22.14	26.91	15.33
MKS-H27	16.10	9.10	15.60	25.02	27.99	6.20
MKS-H29	17.20	7.93	11.92	17.79	33.54	11.62
MKS-H30	14.55	4.20	8.49	16.55	32.78	23.43
MKS-H31	16.45	5.47	10.09	18.68	35.48	13.84
MKS-S1a	46.62	11.55	12.17	13.86	2.81	13.00
MKS-S1b	7.30	10.71	20.97	18.48	38.49	4.05
Mortar Average	14.24	8.21	12.83	20.62	30.70	13.40

Table 8. Granulometrical analysis on the aggregates of mortar and plaster samples

Samples	TB (%)	TA (%)	Samples	TB (%)	TA (%)
MKS-H1	93.63	6.37	MKS-H16	89.93	10.07
MKS-H3	85.57	14.43	MKS-H18	90.37	9.63
MKS-H4	87.94	12.06	MKS-H21	91.77	8.23
MKS-H5	88.04	11.96	MKS-H24	89.46	10.54
MKS-H8	90.54	9.46	MKS-H26	92.24	7.76
MKS-H9	92.41	7.59	MKS-H27	92.83	7.17
MKS-H10	91.82	8.18	MKS-H29	94.49	5.51
MKS-H11	96.63	3.37	MKS-H30	97.97	2.03
MKS-H12	92.98	7.02	MKS-H31	97.93	2.07
MKS-H14	87.45	12.55	MKS-S1a	96.16	3.84
MKS-H15	88.32	11.68	MKS-S1b	75.03	24.97
			Mortar Ave.	91.62	8.38

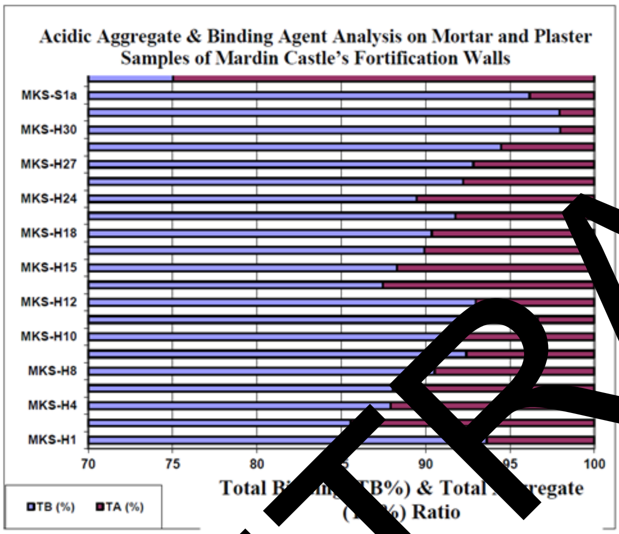

Figure 12. Aggregate and binding agent analysis on mortars and plasters

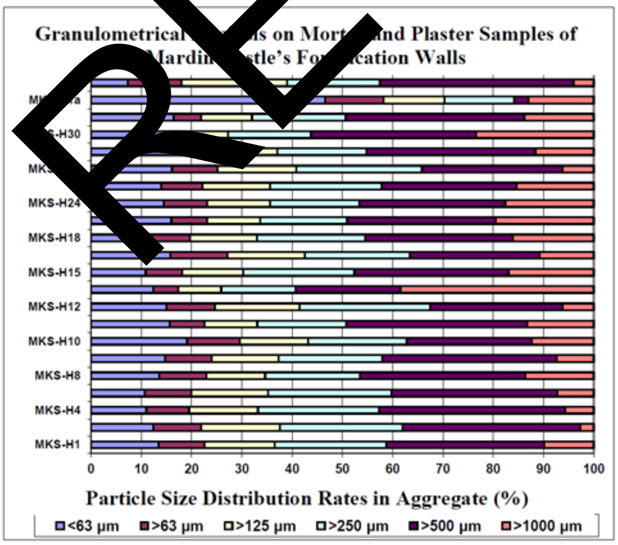

Figure 13. Granulometrical analysis on the aggregates of mortar and plaster samples

Macro-physical structures and particle types of the aggregates obtained after acidic treatment on the mortars of Mardin Castle's Fortification Walls were examined under binocular microscope. It was seen that the macro-physical structures of the aggregates of the samples contained a relatively heterogeneous aggregate length and diversity, which do not have an aggregate type preferred as a result of the certain sieving. It was seen that the aggregate content of the mortars were consisted of the aggregates in rich variety and in compliance with the local formation (limestone), of which the density is rounded.

3.5. Fine Section Optical Microscope Analysis

Thin sections (stone/rock, ceramic (brick and tile) and mortar samples) were prepared so as to show all layers from outside to inside ("Table 7"). Fine sections of the samples were investigated by using LEICA Research Polarizing Microscope D[illegible]P Model optical microscope having bottom and t[illegible] illumi[illegible].

Table 9. Fine Section Optical Microscope Analysis Stone/Rock and Ceramic Samples

Stone Sample Groups	Rock Type	Hardness (Mohs)	Explanations
Stone Gr1a	Biosparitic Limestone	2.5 - 3	Fossil and fossil shells, which are in a nature affected by hydrothermal solutions, are present.
Stone Gr1b	Recrystallized Limestone	2.5 – 3	Contains calcite and dolomite.
Stone Gr1c	Argillaceous Limestone	2.5 – 3	Primarily calcite, and partly quartz and limonite minerals form the structure in silt particle size.
Stone Gr2	Travertine	2.5 - 3	Micro breaks / cracks in the structure, which was formed by hot water precipitation were filled with re-crystallized calcites. In addition, aragonite and limonite are also present in the structure.

Stone Sample Groups
Stone Gr1a : MKS-T5, MKS-T6, MKS-T10, MKS-T14 and MKS-T24
Stone Gr1b : MKS-T1, MKS-T16 and MKS-T17
Stone Gr1c : MKS-T2, MKS-T3, MKS-T4, MKS-T8, MKS-T9, MKS-T11, MKS-T12, MKS-T13, MKS-T18, MKS-T19, MKS-T20, MKS-T21, MKS-T22, MKS-T23 and MKS-T25
Stone Gr2 : MKS-T7 and MKS-T15

3.6. X-Ray Fluorescence (PED-XRF) Analysis

Chemical compositions of stone/rock, brick, soil, lime layer, mortar and plaster samples of Mardin Castle's Fortification Walls were obtained by PED-XRF analysis ("Table 8" and "Fig. 14").

Table 10. PED-XRF analysis values

Element	MKS-T1	MKS-T5	MKS-T8	MKS-T11	MKS-T16	MKS-T20	MKS-T24
Na_2O	0.075	0.080	0.079	0.078	0.088	0.074	0.074
MgO	15.47	0.07	6.76	15.65	15.43	16.70	0.03
Al_2O_3	0.64	0.12	2.15	0.01	0.24	0.04	0.16
SiO_2	4.16	0.52	12.64	0.71	1.57	1.32	0.61
P_2O_5	0.027	0.080	0.054	0.438	0.026	0.160	0.009
SO_3	0.20	0.08	0.17	0.14	3.44	0.22	0.12
Cl	0.045	0.010	0.019	0.049	0.041	0.041	[illegible]
K_2O	0.009	0.007	0.648	0.007	0.009	0.007	[illegible]
CaO	34.80	61.27	40.81	34.10	30.97	[illegible]	61.86
MnO	0.011	0.003	0.020	0.012	0.010	0.015	0.004
Fe_2O_3	0.46	0.06	2.04	0.20	0.22	0.19	0.08
LOI*	44.92	37.91	34.79	48.72	[illegible]	[illegible]	37.82

Element	**MKS-Z1**	**MKS-D1**	**MKS-B1**	**MKS-B2**
Na_2O	0.180	0.073	[illegible]	0.074
MgO	21.34	[illegible]	4.71	4.55
Al_2O_3	0.02	[illegible]	7.58	7.68
SiO_2	0.01	[illegible]	36.90	35.37
P_2O_5	0.074	0.811	0.836	0.835
SO_3	42.39	[illegible]	0.34	0.46
Cl	0.061	0.025	0.020	0.023
K_2O	[illegible]	0.615	2.709	2.933
CaO	[illegible]	34.80	20.74	20.05
MnO	0.002	0.062	0.096	0.107
Fe_2O_3	0.04	2.41	5.74	6.35
LOI*	[illegible]	36.80	19.73	20.93

Chemical composition of mortar and plaster samples of Mardin Castle's Fortification Walls were determined by PED-XRF analysis (''Table [illegible]''). Mortar properties of the samples selected among the mortar and plaster sample groups determined by fine section optical microscope analyses were investigated per aggregate structures. Composition features of the mortar and plaster samples, which were investigated independent from their repair or original qualities, were evaluated by Cementation Index data. Cementation Index (CI) is the ratio of the part dissolved in acid to the part dissolved in bases. Mortars containing lime are classified as fat mortar (FM) and hydraulic mortar (ZHK, OHK and HK) depending on the aggregate content and type. For the mortars, the fat mortars having a total aggregate content less than 5% are the mortars having high levels of lime, thus high levels of CaO (Table 16). Mortars having a total aggregate content more than 5% are the mortars having low CaO levels, thus having hydraulic characteristic. In the composition of this type of mortars rates of silicium (SiO_2), aluminium (Al_2O_3) and iron (Fe_2O_3) are high. Cementation Index (CI) values of the mortars and plasters are provided in 'Table 8'. When the competency properties of the mortar and plaster samples are evaluated, it is seen that the samples contain low amounts of aggregate and have low hydraulic features (''Fig. 14''). Competence of the mortar and plaster samples is very poor and they are close to disintegration.

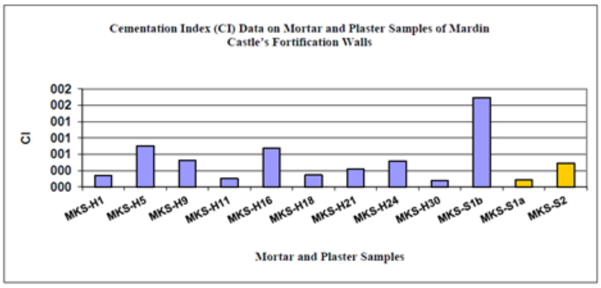

Figure 14. X-Ray Fluorescence (PED-XRF) Analysis Cementation Index Data on Mortars

4. Discussion

Constructional materials of Mardin Castle's Fortification Walls were investigated by various methods.

The first finding that must be emphasized is all samples of Mardin Castle's Fortification Walls are exposed to the effect of high salination. In stone samples, relatively variable and high levels of salination were observed in terms of the average values. The impact of the climatic circle is important at this point (Table 6). When long-term weather conditions of March in Mardin are considered, on average 10.7 days per month is rainy. The heavy rains between December and April in Mardin are relatively higher than the summer season. Average temperature is 8.3°C in March and hours of sunshine are 6.1 hours during the day. Formation of relatively humid environmental conditions were observed in the stone samples, which have been exposed to water accumulation with the heavy side rains, during the sampling carried out in the last period of the winter season, which was rainy and in which the sun showed its face less. This causes an impact, which accelerates the deterioration on the stones. The humid environment formed on the stones causes the limestone stratification, which occurs with the recrystallization of the salines dissolved by lichenification /vegetation on the stone surfaces, with the impact of microclimate, and high salination associated with this, and reveal of destructive effect of salination on the stones (dissolution, disintegration). This finding supports the finding obtained by De Ferri et. al [9] in their studied that water causes disintegration, surface erosion, and cracking through the freezing – dissolution or wetting – drying circles within the pores by acting as a medium for the substances such as sodium and nitrate, and [illegible] the soluble salines, which trigger the crystallization between the pores and dissolution of the stones.

Although it is determined in relatively low amounts in only 2 of the stone samples of the Castle, higher amounts of phosphates were determined in brick, soil, and lime layer samples. In addition, the excessive amounts of various substances such as salines and phosphates show that it supports the finding of Sabbioni et. al. [25] in their studies stating that global warming causes the formation of harmful salines, which affects the porous stones in the entire Central and Northern Europe, and Turkey also supports the finding within this context.

5. Conclusion

In this study, properties of the construction materials and material problems were determined through the studies via spectroscopic and geophysical methods conducted on the materials of Mardin Castle's Fortification Walls. The research is significant in terms of determining the materials and material problems for Mardin Castle's Fortification Walls, which is located in Mardin Province, Turkey, and which survives for centuries as the symbol of the city, and presenting the conservation suggestions. The findings of the study do not only reflect the conservation status of the historical building in real terms, but also provide an experimental base and theoretical support for the conservation of the historical buildings in Turkey. It presents indicative suggestions to establish conservation schemes of the historical buildings and enriches the maintenance and reinforcement evaluation status of the historical buildings.

In the findings, it is seen that salinization is present on the stone samples of Mardin Castle's Fortification Walls, at very variable and high levels, in terms of the average values. This results, which was obtained in the study, match particularly with the findings in the world literature obtained in the studies conducted on the stone structures, stating that the salinization factor is very high [19-24]. It leads to saline crystallization, shell formation, patinas, and alveolization [illegible] pores and cracks. Within this context, primarily it is necessary to prevent the factors, which cause salinization on historical structures.

The original mortar must be conserved on the fortification walls. Lime mortar in compliance with the original mortar content must be used in repairs. In pointing and debris fillings, and in completing the missing parts it is appropriate to use a mixture of 30% lime, 50% ground aggregate, which is sieved, washed, having local (stream bed) material, which does not contain carbonate content, and which the aggregate distribution is in compliance with the original mortars (in an aggregate structure of consisted of 30% on average of rough silt/sand mixture having maximum 1-2 mm of particles and 70% having particles of 63-1000 µm and 2% having particles <63 µm) and aggregate of 15% having silt/clay size and local (containing lime) stream bed material, and 5% of lime mortar containing clay in pozzolanic nature.

It is also possible to use ready-to-use special hydraulic lime, which is produced intended for restoration, in mortar repairs. It is not definitely recommended to use materials containing cement (classic, white or coloured with pigments) in any stage of repair mortar.

The structural stones of the fortification walls are limestone in different sub-types intensely in the research studies. Structural stones are rocks, which belong to the local formation that may be obtained within the vicinity. In repair stage, it is recommended to carry out trial applications for the recommended mortar and plaster contents, and to analyze separately for determining the compliance of the stones/rocks to be selected with the recommended materials.

Acknowledgement

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Conflicts of interest

The authors declare no conflicts of interest.

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