Print ISSN: 2321-6379
Online ISSN: 2321-595X
DOI: 10.17354/ijssl/2017/62

Original Article

Role of Physical and Chemical Analysis of Mortar in Determining Structural Authenticity of Historical Buildings Case study: Choghazanbil Zigorat

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Introduction: Choghazanbil Zigorat is one of the Elamite buildings (13th century BC) with a adobe structure and brick view in southwest of Iran. The building excavated in 1952 AD by Ghirshman has undergone a variety of environmental and human changes and interventions afterwards. Some of these interventions are due to conservation and restoration operations made at the time of Ghirshman, and then, in recent years so that even the main and restored parts of different periods cannot distinguish from each other based on the available evidence.

Materials & Methods: In this study, using statistical analysis of experimental data related to mud mortars used in the building, a model to determine the physical authenticity (origin) of Elamite and restoration parts of Zigorat was provided. The laboratory methods used in the study included X-ray diffraction analyses (XRD), Fourier Transform Infrared (FT-IR) Spectroscopy and combined separation with wet chemical method and grain size distribution of the mortar samples.

Results: The results showed that although sometimes Elamite and restoration mortars have identical compounds and minerals in terms of materials, but their composition ratios and physical properties such as grain size distribution display a very different pattern.

Conclusion: In general, these results suggested that quantitative and qualitative analysis of samples of mortar in historic buildings may play an important role in recognition of the historic and restoration parts.

Key words: Mortar, Elamite, Authenticity, FT-IR, XRD

INTRODUCTION

Choghazanbil complex or religious town of Dur Untash, located at 30 kms from the southeast of Shush in Iran, was founded by the Elam King, Untash Gal in the mid-thirteenth century BC. The town, experienced different building operations only during the 20-year reign of Untash Gal (1245-1265) based on obtained evidence, is mainly damaged in 640 BC by the Assyrian invasions (Grishman, 1995).

According to archaeological reports at Grishman time in 1952, the city consisted of three fences with an area of approximately one hundred hectares that the main temple or Zigorat is located in the center of the first fence. Zigorat, with a square-like plan and each side with 102.20 meter in length has had a height of 52 meters and 5 floors in the past. Today, about 25 meters of the height and two and a half floors have been left of it. The building built entirely of mud brick has been covered with brick façade (Grishman, 1995).

Choghazanbil Zigorat and other architectural remains of this complex after the excavation (prior to the start of the project of conservation and restoration of Choghazanbil in 2000) have been intervened for maintenance in different periods (Ghirshman excavation and later) (Talebian, 2002). Despite the observance of some principles of restoration (difference between the main and restoration parts), some damages and decays have occurred over time. Also, due to lack of necessary documents, recognition of these interventions is difficult in some cases or associated with doubts.[1-6]

Access this article online

IJSS
www.ijss-sn.com

Month of Submission : 06-2017
Month of Peer Review : 06-2017
Month of Acceptance : 07-2017
Month of Publishing : 07-2017

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According to the studies conducted on Zigorat in terms of shape, texture and color of materials, the building construction approach (orientation of adobe and brick, thickness of mortar and grouting made) and comparing the current situation with pictures and documents related to the excavation time and afterwards during the implementation of international project for conservation and restoration of Choghazanbil complex, the historical parts of the building became largely clear and recognizable. However, the physical authenticity of parts of the building were questioned.

How to Determine the Physical Authenticity of Historical-Cultural Works

Authenticity based on Nara Document (1994) about the ability to understand the heritage values depends on the validity and reliability, and as a result, the originality of data resources. Usually, determining the material authenticity of a work is done either directly by its creator with signs of him such as signature and stamp on the work or by using documents like accompanying pictures and texts. Another way to determine the authenticity is indirect by citing to some evidence and analysis of scientific information extracted from the work.

According to the age of some works, one can say that there is not always an access to direct evidence and documents (first method). However, in the second method, determining the time period that the work belongs to with the help of techniques such as archaeological investigations and dating methods, can be a way to determine the authenticity. The techniques and approaches in construction as well as materials used in a work compared with available evidence are as other information that will be useful for determining the authenticity of works (NARA, 1994). Such information can be obtained through laboratory techniques, such as structural analyses, chemical composition, physical and mechanical properties of the construction materials.

Although the Thermoluminescence Dating method is used for determining the age and authenticity of backed materials like bricks used in monuments (Bahrololoomi, 2000), but its results in this case could not be useful because of the same period of all bricks used in Elamite and restoration parts of Zigorat (Talebian, 2002). On the other hand, the presence of brick covering on historical and restoration segments prevented the access to the acceptable number of mudbrick samples used in the building to conduct laboratory studies on this type of materials in Choghazanbil Zigorat and finding interpretable data.

Mortar is of materials that must be made in each period if required and cannot be reused, and its quality may vary from period to period depends on its type, percentage of raw materials and processing. Therefore, a rigorous laboratory study on mortars samples used in different sections of the building associated with statistical data analysis was designed and implemented. The mortars used in this complex (mortar between the adobes and bricks) are mostly made of clay mortars, while in parts of it such as downpipes, plaster mortars and bitumen have been used (Grishman, 1995). This article has discussed the results of mud mortars.

MATERIALS AND METHODS

Heterogeneous nature of traditional mortars requires various macroscopic, microscopic, qualitative and quantitative studies for their true recognition (Middendorf, 2005). On the other hand, since the bulk of mortars used in the Choghazanbil complex is made of mud mortars. It is possible to use the excavated soil (remnants of materials of prior periods) for making new materials, such as mortars in later periods. This may lead to falsification of data and results obtained. Therefore, an increase in the number of sampling, the test error decrease and an accurate interpretation of experiments' data will be obtained.

Sampling

Mortar samples were collected from different parts of Zigorat, wall of the second fences (Table 1). Mortar sampling was carried out during the following steps.

1. Selecting the studied locations separated by each period based on:
    - Examining archaeological documentation and evidence
    - Surveying studied locations and matching with documents and evidence
2. Selecting the location (s) of sampling in the above locations based on:
    - Assessing the environmental situation of the site (in terms of weathering, moisture, etc.)
    - Visual examination of mortar based on texture, color and thickness of the mortar
3. Cleaning and removing surface layers of samples
4. Bulk sampling (as much as possible) with minimal damage to the building
5. Preparation of the sample ID (Hadian, 2008)

Analysis Method

- Identification and evaluation of minerals in the samples using X-ray diffractometer (XRD), SEIFERT 3000 T2T model with copper tube (30 mA and 40 KV)
- Composition identification of samples and salts contained in them by Fourier Transform- Infra Red spectroscopy (FT-IR), NICOLET 510P model
- Quantification of ingredients of mortar samples

Table 1: Profile of mortar samples

Row	Sample code	Mortar type	Period	Sampling location	Geographical situation
1	Ch.Z 78-1/11	Brick Mortar	Elamite	Entrance arch of Inshushinak Temple	Southeast
2	Ch.Z 78-1/12	Brick Mortar	Elamite	End wall of the central hall	Southeast
3	Ch.Z 78-1/13	Brick Mortar	Elamite	Right side staircase wall of the central hall	Southeast
4	Ch.Z 78-1/14	Brick Mortar	Elamite	Left side staircase wall of the central hall	Southeast
5	Ch.Z 78-1/15	Brick Mortar	Elamite	Gallery wall, second floor, staircase right side	Southeast
6	Ch.Z 78-1/16	Brick Mortar	Elamite	Left wall of the porch- like room of staircase	Southwest
7	Ch.Z 78-1/17	Brick Mortar	Elamite	Central hall downpipe of the staircase	Southeast
8	Ch.Z 78-1/18	Brick Mortar	Elamite	Central hall downpipe of the staircase	Southeast
9	Ch.Z 78-1/19	Adobe Mortar	Elamite	Second floor wall, staircase left side	Northwest
10	Ch.Z 78-1/110	Brick Mortar	Elamite	End wall of the stairs- damaged part	Southeast
11	Ch.Z 78-1/111	Brick Mortar	Elamite	Double-sided staircase wall of the layer under brick with inscription	Southwest
12	Ch.Z 78-1/112	Brick Mortar	Elamite	End wall of the stairs- damaged part - Depth of 3 cm	Northeast
13	Ch.Z 78-1/113	Brick Mortar	Elamite	End wall of the stairs- damaged part - Depth of 10 cm	Northeast
14	Ch.Z 78-1/114	Brick Mortar	Elamite	Left side wall, stairs porch room, adjacent to downpipe	Southwest
15	Ch.Z 78-1/115	Adobe Mortar	Elamite	Left side wall, Imperial Gate, second fence	Toward East
16	Ch.Z 78-3/116	Brick Mortar	Elamite	Middle room wall, Imperial Gate, second fence	Toward East
17	Ch.Z 78-1/118	Brick Mortar	Elamite	Gallery wall, first floor, staircase left side	Northwest
18	Ch.Z 78-1/119	Brick Mortar	Elamite	Downpipe internal wall, staircase right side	Southwest
19	Ch.Z 78-1/122	Brick Mortar	Elamite	Staircase wall end, left side	Southeast
20	Ch.Z 78-1/123	Brick Mortar	Elamite	Staircase wall, Floor, Height, Second floor	Southwest
21	Ch.Z 79-1/126	Adobe Mortar	Elamite	Right side of the central staircase, brick wall, second floor	Northeast
22	Ch.Z 79-1/127	Adobe Mortar	Elamite	Right side of the central staircase, brick wall, second floor	Northeast
23	Ch.Z 79-1/128	Adobe Mortar	Elamite	Left side of the central staircase, brick wall, second floor	Northwest
24	Ch.Z 79-1/129	Adobe Mortar	Elamite	Left side of the central staircase, brick wall, second floor	Northwest
25	Ch.Z 79-1/130	Brick Mortar	Restoration	Left side of the central staircase, first floor, first wall	Northeast
26	Ch.Z 79-1/131	Brick Mortar	Restoration	Left side of the central staircase, gallery, first floor	Northeast
27	Ch.Z 79-1/132	Brick Mortar	Restoration	Wall of the left side of the central staircase, first floor	Southwest
28	Ch.Z 79-1/133	Brick Mortar	Restoration	Left side of the central staircase, second floor	Southwest
29	Ch.Z 79-1/134	Brick Mortar	Restoration	Left side of the central staircase, first restoration downpipe	Southwest
30	Ch.Z 79-1/136	Brick Mortar	Restoration (Ghirshman)	Left side of the central staircase, second floor	Southwest
31	Ch.Z 79-1/137	Adobe Mortar	Restoration	End wall of the central staircase, beginning of the third floor	Southwest
32	Ch.Z 79-1/140	Brick Mortar	Restoration	Gallery wall, first floor, northern corner, staircase left side	Northwest
33	Ch.Z 79-1/141	Brick Mortar	Restoration (Ghirshman)	Gallery downpipe adjacent wall, first floor, northern corner, staircase left side	Northwest
34	Ch.Z 79-1/143	Brick Mortar	Restoration	Western corner, staircase right side, first floor	Northwest
35	Ch.Z 79-1/144	Brick Mortar	Restoration	Downpipe adjacent wall, staircase right side, first floor	Northwest
36	Ch.Z 79-1/145	Adobe Mortar	Restoration	Brick wall, northern corner, first floor, staircase left side, second floor	Northwest
37	Ch.Z 79-1/146	Brick Mortar	Restoration	Wall near the first platform, staircase right side	Southeast
38	Ch.Z 79-1/147	Brick Mortar	Restoration (Ghirshman)	Gallery wall, first floor, staircase right side	Southeast
39	Ch.Z 79-1/148	Brick Mortar	Restoration	Inshushinak Temple upper wall, staircase right side	Southeast
40	Ch.Z 79-1/149	Brick Mortar	Restoration (Ghirshman)	Gallery wall, first floor, staircase right side, near the downpipe, southern corner	Southeast
41	Ch.Z 79-1/150	Brick Mortar	Restoration	Gallery wall, first floor, staircase left side	Southeast
42	Ch.Z 79-1/151	Brick Mortar	Restoration (Ghirshman)	Eastern corner, second floor, staircase right side	Southeast
43	Ch.Z 79-1/152	Brick Mortar	Restoration (Ghirshman)	Second floor wall, adjacent to downpipe, Northern corner, staircase right side	Northeast
44	Ch.Z 79-1/153	Brick Mortar	Restoration	Gallery wall, second floor, Northern corner, adjacent to downpipe, staircase right side	Northeast

(quantitative-combinatory isolation) by wet chemical method (Teutonico 1998)

- Grain size distribution of mortars by sieves in gauges 200, 100, 50 and 30 (Teutonico, 1998)

The mortar samples were first classified based on appearance characteristics and field studies into two Elamite and restoration mortars (Ghirshman period and after Ghirshman time). In the following, the results were discussed accordingly and matching with experimental data.

RESULTS & DISCUSSION

Mineralogy (XRD)

The mineralogical results of Elamite and restoration mortars by XRD method are summarized semi-quantitatively in Table 2. As seen in the Table, a group of mortars has two major components of calcite and quartz, and the second group has one major component of gypsum. Other feldspar, dolomite, clay, gypsum and halite minerals have been identified with moderate to low-value variable ratios

Table 2: The results of semi-quantitative mineralogical study of original and restoration mortars samples of Choghazanbil by XRD method

Row	Sample code	Gypsum	Dolomite	Calcite	Clay mineral	Quartz	Feldspar	Halite
1	Ch.Z 78-1/11	++++	+	+				
2	Ch.Z 78-1/12	+	tr	++		++++	Tr	+
3	Ch.Z 78-1/13	+		++		++++	+	Tr
4	Ch.Z 78-1/14		tr	++	Tr	++++	Tr	
5	Ch.Z 78-1/15	+++		++++	Tr	++	Tr	+
6	Ch.Z 78-1/16	+	tr	++++	+	++	Tr	+
7	Ch.Z 78-1/17	+++	+	++++		++++	Tr	
8	Ch.Z 78-1/18	++++	+	+		Tr		
9	Ch.Z 78-1/19			++++	+	++	Tr	
10	Ch.Z 78-1/110	++++		++	Tr	++		
11	Ch.Z 78-1/111		+	++++	+	++		
12	Ch.Z 78-1/112		+++	++	+	++++	+	
13	Ch.Z 78-1/113	+	tr	++++	+	++++	+	
14	Ch.Z 78-1/114		+	++	Tr	++++		
15	Ch.Z 78-1/115			++++	+	+++		
16	Ch.Z 78-3/116	+		++	Tr	++++	+++	
17	Ch.Z 78-1/118		+	++++	Tr	+++		
18	Ch.Z 78-1/119			+		+		+
19	Ch.Z 78-1/122	++++		+++		+		
20	Ch.Z 78-1/123		+++	++++	+++	++	+	
21	Ch.Z 78-1/124		+	++++		++	Tr	
22	Ch.Z 78-1/125		tr	++		++++		
23	Ch.Z 79-1/126		+	++++		++	Tr	
24	Ch.Z 79-1/127		+	++	Tr	++++	+	
25	Ch.Z 79-1/128	++	+	++	+	++++	+++	+++
26	Ch.Z 79-1/129	+++	tr	++++	Tr	++++	Tr	
27	Ch.Z 79-1/130	tr		++++		++	+	
28	Ch.Z 79-1/131		+	++++		++++	+	
29	Ch.Z 79-1/132			++		+++	+	
30	Ch.Z 79-1/133			++		++	+	
31	Ch.Z 79-1/134	+++	+	++		+++		
32	Ch.Z 79-1/136		+	++		++++	+	
33	Ch.Z 79-1/137			++++		++	+	
34	Ch.Z 79-1/140		+	++		++		
35	Ch.Z 79-1/141	++++	+	++		+++		
36	Ch.Z 79-1/143		+	++		++	+	
37	Ch.Z 79-1/144	+	+	++		++	+	
38	Ch.Z 79-1/145			+++		++++	+	
39	Ch.Z 79-1/146		+	++++		++	+	
40	Ch.Z 79-1/147			++		++++	+	
41	Ch.Z 79-1/148	++		++++		++	+	
42	Ch.Z 79-1/149		+	++++		++		
43	Ch.Z 79-1/150			++++		++++	+	
44	Ch.Z 79-1/151	+++	+	++++	+	++	+	
45	Ch.Z 79-1/152			++++		++	+	
46	Ch.Z 79-1/153			++++		++	+	

Very high (++++), High (+++), Moderate (++), Low (+), Trace (tr)

in the first group. In the second group, calcite and dolomite in low levels and sometimes quartz and calcium sulfate with half a water molecule are seen.

In another case (Ch.Z 78- 1/119), halite of salt mineral as the main mineral and quartz and calcite in very low levels have been detected. The presence of this mineral has been also detected in some cases to a lesser extent. With the exception of Ch.Z 78-1/118 located in the northwest side, all are related to two fronts of southwest and southeast. Also, the gypsum as a minor component or impurity, is mostly seen in the main samples of the building in two southwest and southeast fronts. In addition, the presence of clay minerals in restoration mortars (from code Ch.Z 79 – 1/130 onwards) has not been seen in others except one case, code Ch.Z 79- 1/151, while this mineral occurs to be seen in most main cases. It should be noted that large amounts of calcite in the mortar samples sometimes prevent the emergence and detection of peaks related to the clay minerals (Yusuf et al., 2004).

FT-IR spectroscopy

FT-IR analysis results show that the presence of carbonate anions (1441 cm^{-1} area), silicate and quartz (1000-1100 cm^{-1} area and twin peak at 777 cm^{-1} area) are corresponded with the results of mineralogical studies respectively as calcite, clay, feldspar and quartz minerals except for a few cases (Hadian, 2008). However, nitrate ions not detected in the XRD experiment are observed here more or less in all the main samples (1385 cm^{-1} area), and this anion can be seen with low levels in restoration mortars only in three cases, i.e. code Ch.Z 79-1/142., code Ch.Z 79-1/138, and code Ch.Z 79-1/134 (Figures 2 and 3).

The XRD and FT-IR results of a number of samples were compared in the following. In the mineralogical testing, Ch.Z 78-1/110 and Ch.Z 79-1/128 samples of gypsum have been detected as a major component (Table 2). However, the FT-IR analysis does not show the presence of this mineral, and determines its primary ingredients as carbonate, silicate and silica. Samples Ch.Z 78-1/114 and Ch.Z 78-3/115 Seem to be a mixture of plaster and soil by FT-IR testing and have a major component of sulphate (1115, 669, 661, 1622 Cm^{-1}) (Figure 4); in mineralogical study, the presence of gypsum has not been reported in the samples. According to the XRD results, halite is the major and main component of Ch.Z 78-1/119 sample, and calcite and quartz are seen in the case in very low levels; while, in FT-IR testing, the components of carbonate, nitrate, silicate and silica are determined as its main components. It is worth noting that the mineral halite cannot be recognized by infrared spectroscopies. Unlike mineralogy results indicating gypsum as the major and main component of sample Ch.Z 79-1/141, according to FT-IR analysis, the peak intensity of sulfate or gypsum is less than of carbonate and silicate.

Quantitative – Combined Separation

Based on the results of above tests, the components of mortar samples were isolated and measured in four main groups. These four groups included:

1. Lime, including carbonate compounds of calcite and dolomite
2. Gypsum, including gypsum sulfate compounds and calcium sulfate with half a molecule of water
3. Clay, containing very fine insoluble particles (suspended in water), including silica and silicate compounds
4. Sand, containing silicate and silica particles coarser than clay and silt that deposit quickly.

Here, components such as soluble salts of sodium chloride and nitrates were neglected as a component. The results have been shown as diagrams in Figures 5 and 6, respectively as Elamite and restoration mortars and based on the percentage of composition mentioned.

Figure 1: Choghazanbil Zigorat

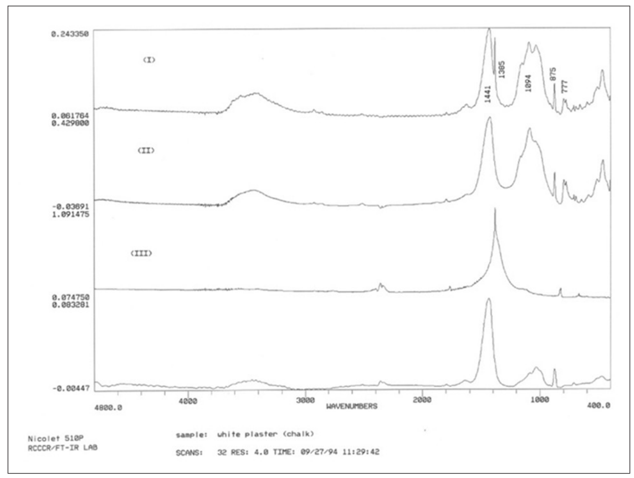

Figure 2: The FT-IR spectrum of Elamite mud mortar (I) compared with control samples: soil (limestone, sandstone) (II), nitrate salt (III), lime (IV)

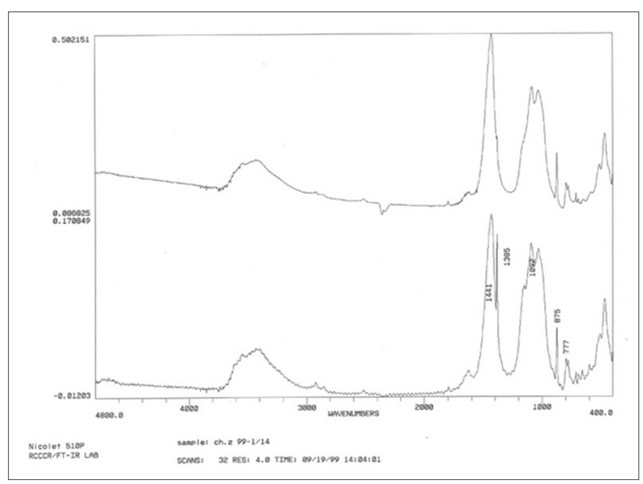

Figure 3: Comparing the FT-IR spectrum of Elamite mud mortar (bottom) and restoration

Elamite Mortars

As seen in Figure 3, percentage variation of gypsum in these samples is between 1-5 %. Regardless of samples

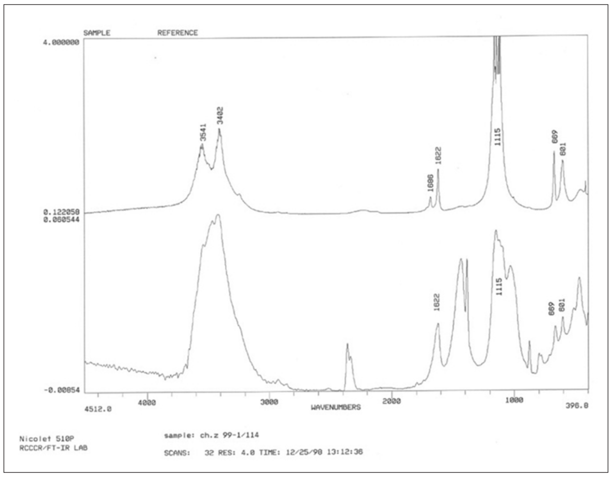

Figure 4: FT-IR spectra of Elamite mortar Ch.Z 78-1/114 (bottom) and a control sample of plaster (above)

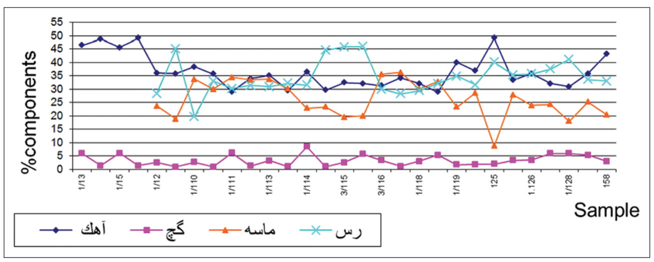

Figure 5: Graph of Elamite mud mortar ingredients – Choghazanbil, Dark blue: Calcite, Purpale: Gypsum, Orange: Sand, light blue: Clay

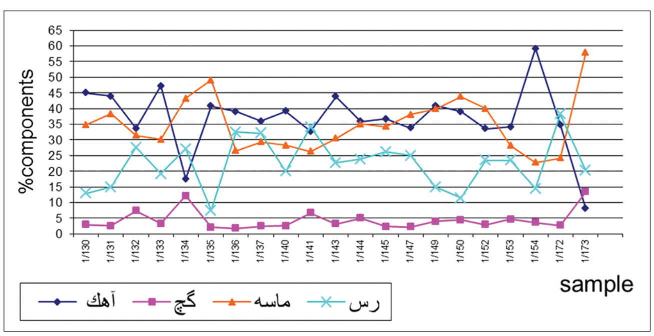

Figure 6: Graph of Restoration mud mortar ingredients – Choghazanbil, Dark blue: Calcite, Purpale: Gypsum, Orange: Sand, light blue: Clay

1/13, 1/15 and 1/16, which could not be separated completely due to the low quantity of samples, in most cases, the percentage of lime is around 30-35%.

The clay content in the samples were divided into two groups. Some of them in the vicinity of 30-35%, and a number, mostly related to the mortars between the bricks, are above 40%. An exception is in the case 1.110 with the amount of clay much less than similar samples. This sample was related to the damaged section of the wall.

The percentage of sand was also divided in two groups: A group with about 35-30% and the second group with less than 25%. Two different results were obtained in repeating the test on sample 1.114. This sample also showed different results in FT-IR and XRD analyses, and as previously mentioned, this sample is related to the wall adjacent to downpipe, which has been highly affected by water washing, resulting in changes in the percentages of its ingredients in different parts. In general, two patterns of compositional analysis have been obtained for Choghazanbil samples. In pattern 1, the proportions of clay, sand and lime are close to each other as 30-35%, while in pattern 2, the clay content is about 40% and above, and correspondingly, the sand content lowers; but, the range of limestone is almost as 30-35% trend.

Restoration Mortars

A glimpse into the charts show that two Elamite and Restoration groups of mortar have general and distinguishable differences. As seen in Figure 5, the ranges of lime and sand changes in restoration mortars compared to the original mortars have a higher level, while the range clay variation is in the lower level. Also, as seen, the graph fluctuations of restoration mortars are much more than Elamite mortars, which is expected because of the diversity restorations carried out in the building. In some cases like samples 1/132, 1/136, 1/137 and 1/141, the ratios of combined components are close to the Elamite samples.

In overall, the range of percentage of gypsum (sulphate compounds) in these samples are mainly between 1-5%. The variation range of clay in most cases is about 25% and lower, while the amount of lime is about 35% and more.

Grain Size Distribution

After separation of ingredients of the above mortars, their sand portion were graded by a series of sieves in particle size of 200, 100, 50 and 30. The percentages retained on the sieves for samples were compared by using graphs and statistical analyzes. The samples are discussed here in two Elamite and Restoration groups.

Elamite Mortars

As can be seen from the bar graph for each of the samples (Figure 6), Choghazanbil samples graining (grading) follow two general patterns. A series of samples, such as 1/12, 1/19 and 1/112 have a peak at mesh 200, and the second series like 1/111, 1/118 and 1/127 have two peaks, one in mesh 200 and another at mesh 30, where the former is taller than the latter.

Restoration Mortars

Comparing the grading pattern of main and restoration samples at first glance, one can see that in some cases, the pattern of restoration and Elamite mortars are similar, while in other cases, the grading of restoration mortars are much larger. In total, grading patterns mainly determined for this group of mortars are in two types (Figure 7).

1. A peak at mesh 200
2. Two peaks, which is taller at mesh 30 than mesh 200, such as: 1.131, 1.140, 1.141, 1.144, and 1.153.

It should be noted that due to low sample size, was not possible to do hydrometric testing for grading of clay portion.

In total, the portion of fine- grained raw mortars (Elamite) is greater than restoration mortars.

CONCLUSION

The results obtained from experimental studies on Choghazanbil Zigorat mortars are summarized in the following sections:

The mortars used in the rows of bricks and adobes in the main sections are made of two types of clay with fine-grained dolomitic limestone, and sometimes with brick debris. The main minerals forming these soils include calcite and quartz in high proportions, dolomite and clay mineral in low levels, feldspar and gypsum in variable amounts from moderate to low levels. The presence of absorption band of nitrate in the FT-IR spectrum of all Elamite samples is considered as a fingerprint to distinguish them from the restoration mortars.

The percentages of the ingredients of clay, sand, lime and plaster (headings defined in the quantitative - combined section analysis) for soil type 1 were as follows: 30-35%, 30-35%, 30-35% and 1-5%, while type 2 contained clay over 40%, sand less than 25% and limestone about 30-35%. The changes in the amount of gypsum are the same 1-5%. The second type can be seen more in mortars between the adobes. The grading of sandy section of these soils also follows two different patterns. The

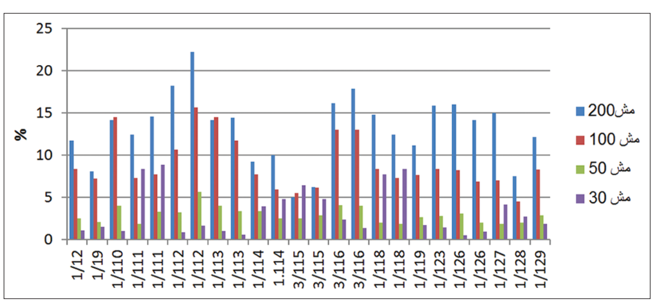

Figure 7: Graph of Grain size distribution of sandy portion of clay mortars - Elamite Choghazanbil, Mesh: 200, 100, 50, 30

remaining fine-grain portion of these sands on the sieves 100 and 200 is much higher than those retained on the sieves 30 and 50.

The mortars used between adobes and bricks in the restoration parts are more diverse. The mineralogical results of these mortars show no large differences with Elamite mortars, except that clay minerals are rarely seen among them. However, the presence of a large percentage calcite in the sample usually prevents the emergence and detection of absorption bands related to the clay minerals in the XRD spectra. In these samples, there is no sign of the peak related to nitrates in FT-IR spectrum, which distinguishes them from the original samples. Mineral halite is not seen in any of the samples, and gypsum is also found variably and mostly in the amount of 1-5%. The quantitative pattern of the restoration mortars ingredients of Ghirshman period is close to the main samples, but in samples after the Ghirshman period, the clay content is less than 25%, while the lime rate is over 35% and sand content varies between 20% and 50%.

Comparison of quantitative analysis, gradation and qualitative analysis of a number of samples, which their periods had been estimated based on appearance characteristics, do not match with the experimental results. These examples are as follows:

- Sample Ch.Z 79-1/135 which is related to the "Sofeh" wall of the first floor, left side of the central staircase in the southwestern side front, and detected as the mortars of Ghirshman period. Its results are similar to those samples after the Ghirshman period.
- Sample Ch.Z 79-1/149, which is the mortar between the adobes and related to the brick wall on the second floor in the northwest side. It has the composition and size distribution of Ghirshman period mortars. This mortar had been classified as mortars after the Ghirshman period, and vice versa, in the case of Ch.Z 79-1/149 considered as mortars of Ghirshman period, there is no similarity in composition analysis with mortars during this period.
- Samples 79-1/152 Ch.Z and 79-1/153 Ch.Z have both similar compounds with mortars after the Ghirshman period, whereas the first mortar had been determined belonging to the Ghirshman period on visual examination.
- Samples Ch.Z 79-1/136 and Ch.Z 79-1/137 both have composition and grain size distribution similar to the Ghirshman period mortars, while the mortar sample of Ch.Z 79-1/137 had been determined as mortars after the Ghirshman period.

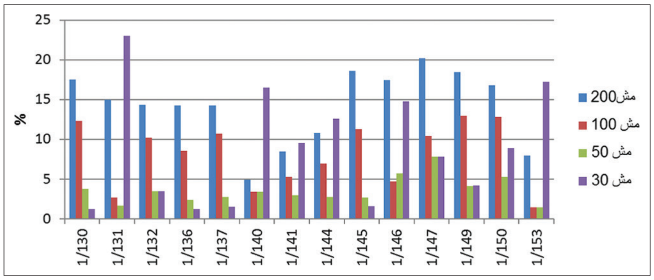

Figure 8. Graph of Grain size distribution of sandy portion of clay mortars - Restoration Choghazanbil, Mes h: 200, 100, 50, 30

Therefore, the site of above mentioned samples needs to be re-studied in terms of historical period to ensure their authenticity according to the results of tests conducted. (Table 1-2, Figure 1-8)

ACKNOWLEDGEMENT

At the end, we would like to sincerely appreciate the support and guidance of Dr. Rasool Vatandoost, and Dr. Mohammad Hassan Talebianas well as the cooperation of Mrs. Soheila Zakavi, in performing wet chemistry experiments in Haft Tapeh Research Center, Mrs. Soodabeh Dorali for doing X-ray diffraction test in the Conservation and Restoration of Cultural Relics (RCCCR), and Mr. Bijan Heidarzadeh, and Mr. Hossein Hosseini for their cooperation in sampling.

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How to cite this article: Dehkordi H, Manijeh, Madani, Sadat F. Role of Physical and Chemical Analysis of Mortar in Determining Structural Authenticity of Historical Buildings Case study: Choghazanbil Zigorat. Int J Sci Stud 2017;5(4):448-455.

Source of Support: Nil, **Conflict of Interest:** None declared.