Study and Recognition of Mineral Matter of Pastiglia Layer and Gliding of Safavid Period in Qazvin Chehelsotoun

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Abstract

The wall paintings of Qazvin Chehelsotoun Mansion include two periods in the early Safavid period and one period in the Qajar period. This topic has received little attention in the field of structural studies and stylistics. Investigating these decorations in order to understand the evolution of this art and considering the temporal precedence over other murals in the Safavid period seems necessary. (Especially in Isfahan) The main question of this research is to identify the structure and examine the differences and similarities between the techniques of layering and gilding in different layers related to the Safavid era in this monument. The research hypothesis is based on the similarity of materials and techniques used in the two short and successive time periods. The purpose of the study is to identify possible differences in the structure of the materials and techniques of layering and gilding in these two eras of the Chehelsotoun monument. For the reasons mentioned above, the western part of the south corridor and the motif patterns associated with these two periods were selected. Dental instruments were used for sampling to the depth of the plaster bed layer. After microscopic examination and coding of the samples, two specimens were selected to study the motif layers of both periods from the upper to the lower substrate. To identify the elements and ingredients of these motifs, the microscopic images were examined by SEM-EDX microscope.

In order to identify and detect the composition of materials in different phases, different decorative and gilding layers (gold layer, red soil layer and plaster substrate) were examined by XRD imaging. The results of this research show that the techniques of layering and gilding are identical in these two periods. There is only a slight difference in the purity of the gold and plaster substrates used during these periods. The gold used in the first period has metallic impurities and its gypsum substrate contains the anhydrite phase which is not seen in the second period. The results can be useful in studying the art of mural painting. These findings can be used for further studies regarding the recognition of clamps and preservatives material used in building decoration.

Keywords: SEM-EDS, XRD, Pastiglia, Gilding, Qazvin Chehelsotoun.

Introduction

The city of Qazvin is located in the northern part of Iran and west of its capital. Qazvin has been the capital city of Iran for more than half a century in the Safavid era.

Today only a few important monuments of that era remained. That is why they all need special attention to identify, maintain and introduce.

Among these monuments, the Chehelsotoun mansion is very important because of the remaining of the early Safavid murals.

For this reason, this monument and its decorations require extensive research (Figure 1).

Wall paintings are among the decorative arts used in architecture that incorporate any type of paintings with layers of color.

Based on the quality of the work, this art consists of at least two layers, including of support (architectural structure) and color layer (motifs) and can extended up to six layers. These layers are: supporter, primer layer, bedding layer, canvas painting layer, color layer and protective or varnish layer (Hamzavi, 2016).

Wall decorations of Qazvin Chehelsotoun mansion have been created in three time periods. These include two periods in the early Safavid kingdom and one in the Qajar period. (Probably during the reign of Saad al-Saltanah)

Many scholars believe that the first layer of wall paintings and ornaments of the Chehelsotoun was built during the reign of Shah Tahmasb Safavid. (Mashrouteh, 2012: 75; Monshi e Qomi, 1987)

The second layer of these decorations seems to be from the time of Shah Abbas Safavi. (Parhizgari, 2012: 489).

◄ **Fig. 1. Location of Qazvin Province in Iran and pictures of Qazvin Chehelsotoun Mansion (Authors, 2019).**

The question of this study is to identify and investigate the differences and similarities between the layered arrays and the gilding of the Safavid era and The hypothesis of this study is that, the techniques of work execution in these two periods are similar. Of course, differences in the style of the mural of these two periods can be seen due to the change in artists and their styles.

These differences can be identified by studying the style of the murals. The purpose of this study is not to investigate the style of the motifs. Rather, structural recognition and consideration of possible variables in the technique of placing the layers and gilded in those two periods are considered.

Research Background

In 1544 AD, during the reign of Shah Tahmasb, due to the political and military conditions of this time, he provided the arrangement for the change of the capital city from Tabriz, which located in northwestern Iran. According to historical sources, he officially chose Qazvin as the capital city in 1555 AD. (Ghazi ahmad 1980 - Yarahmadi, Ansari, & Mahdavinejad, 2018: 28)

The royal garden in the city (Saadat Abad) was built as a government center by the engineers of that time with a detailed plan. It was dedicated to the royal family, courtiers and guests. All of these works were beautifully and artistically made.

Today, there are only two monuments left from that large and 74-hectare complex: Aliqapoo gate and Chehelsotoun Mansion.

The Chehelsotoun or Pavilion Mansion, known in the Safavid era as the Arshikhaneh and Hasht behesht (Eight Paradise), has received many names throughout history (parhizgari, 2012: 487-489). The mansion is located in the northern part of the State Complex and is being built at the intersection of main streets.

The main part of the building was built on a single floor during the reign of Shah Tahmasb. This section has an octagonal plan and interior and exterior painting decorations.

The second floor was probably added to the building during the reign of Shah Abbas. The decoration of this section is similar to the style of painting used in the second period of the Safavid dynasty. (Mashrooteh, 2012: 40; Parhizgari, 2012: 489).

The monument has been repaired in various eras. Over time, many extensions have also been added to the building. (Table 1)

The building was listed in the National Register of heritages in the winter of 1956. (With registration number 389) The national registration of the building made it better to protect and maintain it. Chehelston Historical Complex has been in use as a museum since 1998. (Qureshi, 2013: 69).

Although the murals in Qazvin Chehelsotoun's monument are important in understanding the painting schools, and in particular the Qazvin school, some scholars attribute some of these paintings to Shah Tahmasb himself. This increases the importance of these paintings.

Unfortunately, there is no study of the structure of these paintings so far. Previous studies have focused more on the introduction, construction, repairs, extensions, restoration and decoration of the building on the basis of historical documents and evidences left over. (Yarahmadi, Ansari, & Mahdavinejad, 2018, Monshi e qomi 1986, Dabir Siaghi 2002, pazooki 2005, Eshraghi 2009, Parhizgari 2012, Mashrooteh 2012, Ghoreishi 2013)

Of course, researchers have studied the ornamentation of layers and gilding in other Safavid monuments and other pre and post Safavid eras.

The results of a research paper published by the researchers, examined the construction techniques and elements used in decorative layers and the use of gold in some of the most famous monuments of the Safavid era, such as the Ali Ghapou Palace, Chehelsotoun and Hasht Behesht of Isfahan.

In the study described above, experiments and analyzes were

Table 1. Intervention Periods (Reconstruction, Repair, Restitution, Extensions) in Qazvin Chehelsotoun (Parhizgari, 2012: 489; Mashrooteh, 2012: 40; Jabbari and Fathi, 2008: 74).▼

Type of intervention in Qazvin Chehelston mansion	Teriod(s)
changes in the elements of government place (Aowlat Khaneh) and garden of Aaadat Abad	Safavid period, king Ismail ii
add the second floor and its stairs from the southeast direction of the mansion	Safavid period, Shah Abbas (1629 ad)
the first restoration of the building	Qajar period (1878 ad)
second period of repairs, especially on the second floor and elevation of the roof, brick fitting with semicircular arch around the building, and construction of stairs for the second floor on the north side	the Gajar period (1889-1888), during the reign of Saad al-saltanah
third period of restoration	the qajar period (1902 ad), Salar akram's reign
build a staircase for the mansion	the Qajar period 1919 to 1920a.d - reign of Nezam al-sultan and Mufafagh al-dawlah
the stairs were removed and demolished	The Qajar period (192 a.d)
paintings found on gypsum substrate - second floor access path changed - galvanized sheet gable roof installed	Pahlavi period (1957 a.d)
the first scientific reconstruction of the building was carried out - tie stiffening and strengthening was done.	Pahlavi period (1968)
the paintings were peeled and some paintings were removed from the building	Pahlavi period (1973)
the tie wrapping was done around the porch	Pahlavi period (1978)
all preservation and restoration activities were stopped	Islamic republic period (1981)
instead of wooden columns, iron beams were inserted at the base of the columns for damping and moisture removal, channeling was performed.	Islamic republic period (1982)
the roof of the building were repaired.	Islamic republic period (1992)
this monument became a museum.	Islamic republic period (1998)

performed by using the SEM-EDX, TLC, and XRD methods. In these analyzes, the composition of the studied decorative layers was identified, including unassisted plaster, Armenian mud and a type of protein adhesive. (Holakooei, Abed-esfahani, & Aslani, 2013)

Another article examines this issue as a gilding study of molded arrays made of gypsum in Seyed roknal-Din in Yazd. In this study, a specific type of gilding has been identified and introduced using a tin plate with a transparent yellow coating in the Al-e-Muzaffar period. (Hamzavi and others, 2013).

In an article entitled "Architectural Arrangements of the Tomb Room of Pir e bakran Historical Monument and its Hidden Inscriptions", studies on gilding on gypsum inscriptions of the Ilkhanid period have been carried out (Hamzavi and Moazeni, 2013).

In an article titled "Discovering Ancient Inscriptions at Vanak Church in Isfahan", the researchers examined the arrays in the decorative and gilded layers of the building. In terms of quantity, this church is one of the buildings with the largest volume of decorative and gilded layers in Iran (Hamzavi, 2014).

In an article entitled "Recognition and Restoration of the Inscription Layers of the Eastern Backyard (Eastern Krias) of the Great Mosque of Yazd" the use of the gold sheet in the inscriptions of the mosque has been identified and the decorative murals of the historical tomb of Seti Fatim in Yazd have been studied. It is known that the monument dates back to Timurid period. (Hamzavi and others, 2015).

In an article titled "Grafting Technique on Stone at Golestan Palace Complex in Tehran", SEM-EDS and FTIR analytical methods have been identified using oil-based grafting techniques(possibly bow oil). (Ahmadi, Abed Esfahani, and Mohtasham, 2011).

In another article entitled "Technical Study of Gilding Decoration in Stone inscriptions of Vank Church in Isfahan", by using SEM-EDS and FTIR Analytical Methods, The gold-plating technique with gold sheets with an oil interface has been introduced (Mohtasham, 2016).

In another article entitled "Technique and Investigation of the Mechanism of interfaces in Decorative Layers Implemented on Stone during Qajar period" by XRD, SEM and FTIR analysis methods, it was found that live gypsum with probable asphodel material are two important factors in bonding. These materials provide the necessary bonding and connections between the decorative layers and the base surface. (Mohtasham, Ahmadi and Abed Esfahani, 2010).

"Technical Study of Plaster Decorations of Shahshan House in

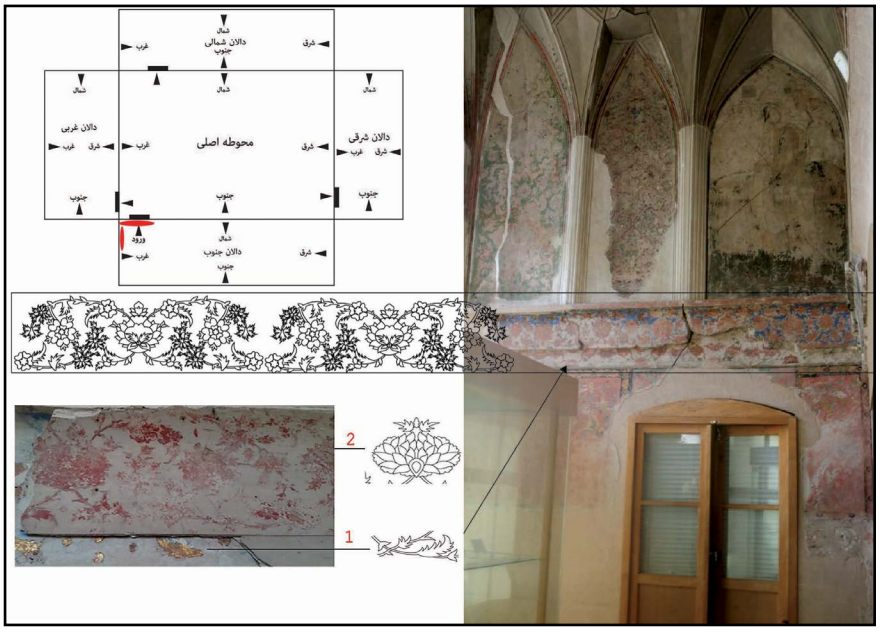
Isfahan from the Qajar Period" is the title of another article that has done valuable research in this area. In this research, SEM-EDS and XRD study methods were used.

Accordingly, it was found that the thatch lining or Bottom layer, two plaster beds and plaster ornamentation using semi-mortar plaster were used to carry out these decorations. (Mohtasham and others, 2014).

Materials and Laboratory Methods

Initially, gilded ornaments and remains decorations on the layers of the Qazvin Chehelstone Mansion in the western part of the south corridor of the building were selected. There were two periods of decoration in this section.

Sampling was carryout with the help of dental and surgical blade to the depth of the plaster bed layer. (Figure 2)



◄ **Fig. 2. Views of the sampling site from the western part of the South Corridor (Authors, 2019).**

Visual observations indicate there are three layers of gold, red layer (red mud) and white layer (plaster) in both samples. The specimens were imaged with the "Dinolite AM73915MZT" digital system.

Subsequently, the samples were photographed and elemental analysis by Scanning Electron Microscopy Analyzer (SEM-EDS).

Initially, the experiments were conducted in the Department of Electrical and Computer Engineering at Tarbiat Modarres University. For the SEM-EDS analyzes, the Phenom ProX device from the Netherlands was used.

The experiments were also performed at the University of Tehran by using the Hitachi s4160-edax amatek at the Faculty of Electrical Engineering.

A third similar experiment was conducted with the QUANTA 450- edax XFlash 6I10 BRUKER at the University of Tehran's Metallurgy Department.

In EDS elemental analysis, the layers usually cling to each other and represent a percentage of the other elements in their layers.

For this reason, in order to better understand the gilded decorative layers implemented in two layers of decorations related to the Safavid period of Qazvin Chehelsotoun, each specimen was selected so that all three layers could be analyzed together.

These samples were tested by surface analysis of each layer. Thus, the increase or decrease of the elements in each layer and the concentration of each element in the specific layers were determined.

The XRD analyzer and the Philips X'Pert MPD diffractometer were used to achieve the composition of the material phases in different layers of the samples. (With Specifications: Tube Co; Voltage 40 kV; Current 30 mA). This experiment was conducted in the laboratory of the Department of Basic Sciences of Tarbiat Modarres University.

The data obtained from this experiment were interpreted by using Xpert high score plus software.

In this experimental method, unlike EDS elemental analysis, the layers of each period were individually examined and the identification of the constituents of each layer was performed more precisely (Table 2).

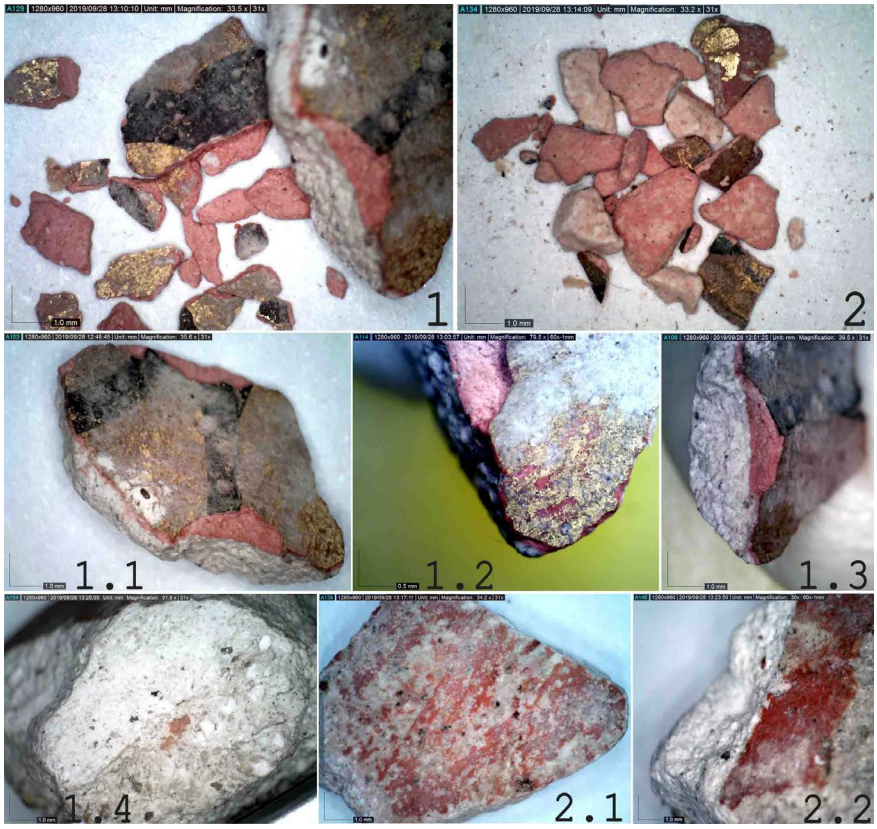
Table 2. Coding of Selected Samples from the Safavid Period in Chehelsotoun Mansion (Authors, 2019). ▶

Historical period (estimated)	Type of analysis	Sample numbers	Golden layer	Red layer	White layer
Shah Tahmasb	SEM-EDX	N.1	N.1.1	N.1.2	N.1.3
Shah Abbas		N.2	N.2.1	N.2.2	N.2.3
Shah Tahmasb	XRD	X.1	X.1.1	X.1.2	X.1.3
Shah Abbas		X.2	X.2.1	X.2.2	X.2.3

Results and Findings of the Research

In studies with Digital Loupe on samples from both periods, three layers were identified. These layers, respectively, from outer to inner part are: gold layer, red layer and white substrate

To the naked eye, these layers are also visible in the fracture sections of the monuments. On some samples, parts of the red layer are thicker, which makes that parts of painting thicker than others. Also, in some samples there are two layers of white (plaster) of varying thickness, the lower layer being relatively darker and probably is the lining base (Figure 3).



◄ **Fig. 3. Microscopic Images of Layers and Gilding Samples of Qazvin Chehelsotoun Mansion in the Safavid Period. Figures 1 to 1.4 relate to the first period and show gold, red, base (substrate) and liner layers - Figures 2 to 2.2 relate to the second period and show the gold, red and substrate layers (Authors, 2019).**

SEM and EDS analyzes

Sample N.1 is related to the period of Shah Tahmasb and the time of construction.

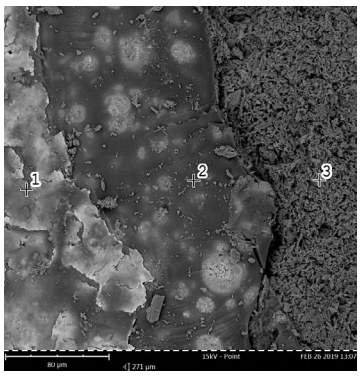
The remains of this period paintings show more gilded ornamentation than the N.2 specimen which probably made during the reign of Shah Abbas.

Table 2 shows the elemental analyzes of N.1 as normalized. It should be said that oxygen and carbon are not included in this analysis.

Sample N.2 was analyzed by the same method as Sample 1. (Table 3).

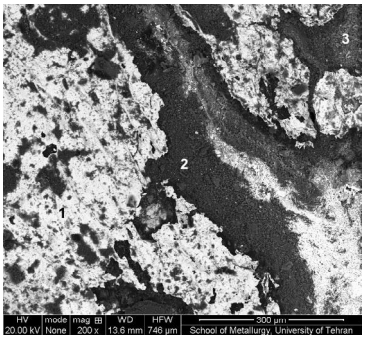
SEM imaging was performed on both transverse sections for close observation of the layers. In these images, the white horizontal line belongs to the surface of the gold layer. The thicker layer is darker in

Table 2. Elemental Surface Analysis of the Layers in Sample N.1 (Authors, 2019). ▶



Elements (Ingredients)	Golden layer (N.1.1)	Red layer (N.1.2)	White layer (N.1.3)
Ca	22.8	51	53.3
S	29.5	35.8	46.7
Si	3.8	5.2	-
Fe	4.8	5.2	-
Al	1.6	2.5	-
K	-	-	-
Na	-	-	-
Au	23	-	-
Cu	6.7	-	-
Zn	6.2	-	-
Sn	1.1	-	-
Mg	-	-	-

Table 3. Elemental Surface Analysis of the Layers in Sample N.2 (Authors, 2019). ▶



Elements (Ingredients)	Golden layer (N.2.1)	Red layer (N.2.2)	White layer (N.2.3)
Ca	34.4	47.9	57.4
S	19	38.5	37.1
Si	0.5	4.7	2.1
Fe	-	4.2	0.3
Al	1.2	2.8	0.5
K	-	1.6	0.9
Na	-	-	-
Au	44	-	-
Cu	-	-	-
Zn	-	-	-
Sn	-	-	-
Mg	0.7	-	0.1

color and more compact in density.This layer is below the gold layer and corresponds to the red part. Below this layer, there is a different and thicker texture that corresponds to the white substrate. In both cases, the gold layer is the thinnest layer (Figure 3).

XRD analysis

In order to increase the accuracy of the results and to reduce the

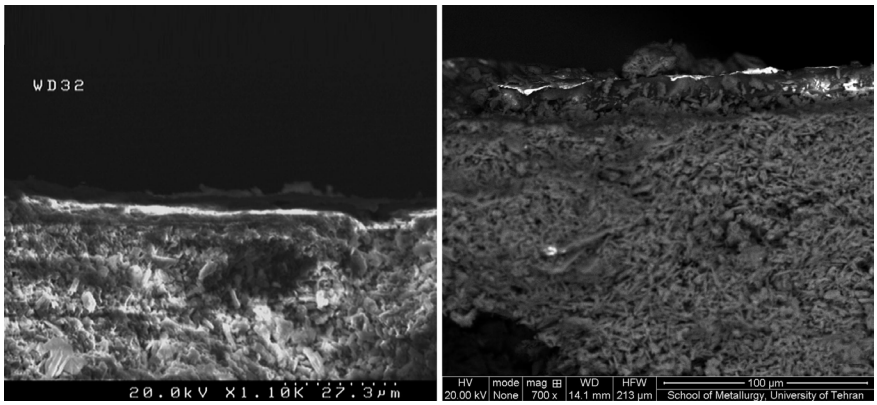


Fig. 3. The left side of sample N.1; the right side of sample N.2 (Authors, 2019). ▶

amount and percentage of elemental mixing in the various layers studied (golden, red and white), the layers of both samples were analyzed separately by XRD.

The results of the analysis of three different layers of plaster substrate, red soil layer and gold layer are presented in Table 4.

◄ **Table 4. Phases identified by XRD (Authors, 2019).**

Sample	NO.	Compound Name	Chemical Formula
X.1	X.1.1	Gold; Gypsum; Muscovite	Au; $Ca(SO_4)$ $(H_2O)_2$; $CaSO_42H_2O$
	X.1.2	Gypsum; Muscovite	$CaSO_42H_2O$; (K, Na)(Al, Mg, Fe)2 (Si 3.1 Al 0.9) O10 (OH)2
	X.1.3	Gypsum; Quartz; Anhydrite	$CaSO_42H_2O$; SiO_2; $Ca(SO_4)$
X.2	X.2.1	Gold; Gypsum; Quartz; Muscovite	Au; $Ca(SO4)(H2O)_2$; $CaSO_42H_2O$; SiO_2; (K, Na)(Al, Mg, Fe)2 (Si 3.1 Al 0.9) O10 (OH)2
	X.2.2	Gypsum; Quartz; Muscovite	$CaSO_42H_2O$; SiO_2;(K, Na)(Al, Mg, Fe)2 (Si 3.1 Al 0.9) O10 (OH)2
	X.2.3	Gypsum; Quartz	CaSO42H2O; SiO_2

Discuss the results and findings

Microscopic examination of samples taken from the Safavid period in the Qazvin Chehelston masonry revealed the presence of different layers of gold, red soil, substrate and base liner. Gold is the thinnest layer and the thickest layer is the base. The base thickness is approximately 5 mm.

The thickness of the red soil layer varies depending on the level of surface highlighting. This layer is about 1 mm in the selected areas sampled in this study.

The second period (N.2) had no base liner layer but in the first period (N.1) the liner layer was observed with differences in color and texture relative to the substrate layer.

In SEM-EDS experiments, the sampling strategy was to study all three layers together. The results of this analysis clearly indicate the concentration of the elements in the layers on top of each other.

For example, elements such as calcium (Ca) and sulfur (S) in layers N.1.3 and N.2.3 which are related to the gypsum substrate

compared to other layers, especially in gold (N.1.1, N.2.1) layers, have more weight.

In layers N.1.2 and N.2.2 the percentage of iron is higher than other layers. This indicates the concentration of this element in the area.

Also, the remarkable presence of calcium (Ca) and sulfur (S) in these layers can be a consequence of the composition of the layers and not merely the overlap of the substrates. In other words, the composition of these layers can be a mixture of gypsum and an iron-bearing mineral.

The red soil layer, which ranges from pink to red in the frescoes, is a kind of filler for the gilding. Elemental analysis of the gold layers in the two periods studied (N.1.1, N.2.1) revealed a slight difference in gold purity.

In layer N.1.1, a significant percentage of the metallic elements of copper (Cu), zinc (Zn) and slightly tin (Sn) are seen, which are related to the impurities associated with the gold sheet.

It should be noted that in the compounds of layer N.2.1 no such metal elements are observed, indicating the purity of the gold sheet in the second period of decoration.

The presence of silicon (Si), aluminum (Al), potassium (K) and magnesium (Mg) can be attributed to the impurity or presence of compounds in the substrate and red soil layers.

In the microscopic images, the gold layer is seen as an integrated coating indicating the use of gold sheets. In contrast, gold pigment images are in the form of powders and dense particles. Microscopic images (Figure 3) show crystals with parallel, deformed, short surfaces with empty spaces. This can indicate excessive kneading and rubbing in the process. In this image, the bright line on the surface corresponds to the gold layer. The underlying dark layer, which differs greatly in thickness, has a uniform shape compared to the underside (plaster bed), which represents the red soil layer.

The XRD experiments also show the accuracy of the results of previous experiments. Layers X.1.1 and X.2.1, except for the gold phase, show phases of gypsum and Moscovium that belong to the red soil layer.

The pink soil layer can be a mixture of gypsum and clay (Illite). Illite is a clay mineral derived from the decomposition of mica and feldspar. Biotite minerals (mica- a black, dark brown, or greenish black variety of mica, occurring in many igneous and metamorphic rocks.) contain iron and potassium in their structure, decomposed by weathering and absorption of water, releasing iron ions and

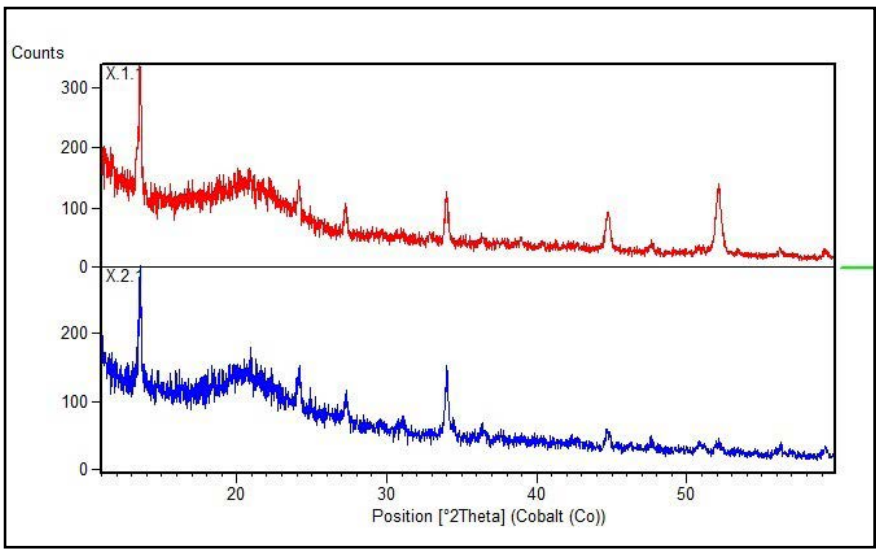
producing a pale pink and reddish color. In addition to iron from biotite decomposition, the muscovium mineral is also produced.

Here, although there is no separate iron element such as hematite and magnetite but it is found in the biotite structure of iron ore. Therefore, the muscovium phase can be related to clay minerals and illite.

In the study of these two different periods, the substrate layers (X.1.3 and X.2.3) have distinct differences. Layer X.1.3 represents the anhydrite mineral that is not present in Layer X.2.3

The presence of anhydrite in this layer can be due to the high temperature of the plaster baking or due to the presence of some salts such as chlorine. These materials produce anhydrite phases when moisture and temperature are present in the gypsum.

In general, the X.1.1 and X.2.1 layers can be considered as the gold phase, the X.1.2 and X.2.2 layers as a mixture of gypsum and illite, and the X.1.3 and X.2.3 layers as gypsum substrates. Comparison of the phases of similar layers in the two wall decorations of the two periods of the Safavid era in Chehelston Qazvin Palace reveals considerable similarities. According to the phases identified, the same compounds are found in the gold layers (X.1.1 and X.2.1) with a slight difference in X.1.1 (Figure 5).



◀ **Fig. 5. Phase comparison chart of the two gold samples (Authors, 2019).**

Comparative diagrams of the phases of the other layers (red soil and gypsum bedding) also show significant similarities and adaptations.

This may indicate the use of the same materials and methods in the two periods of decorating the Qazvin Chehelstone monument in the Safavid period. (Figure 6)

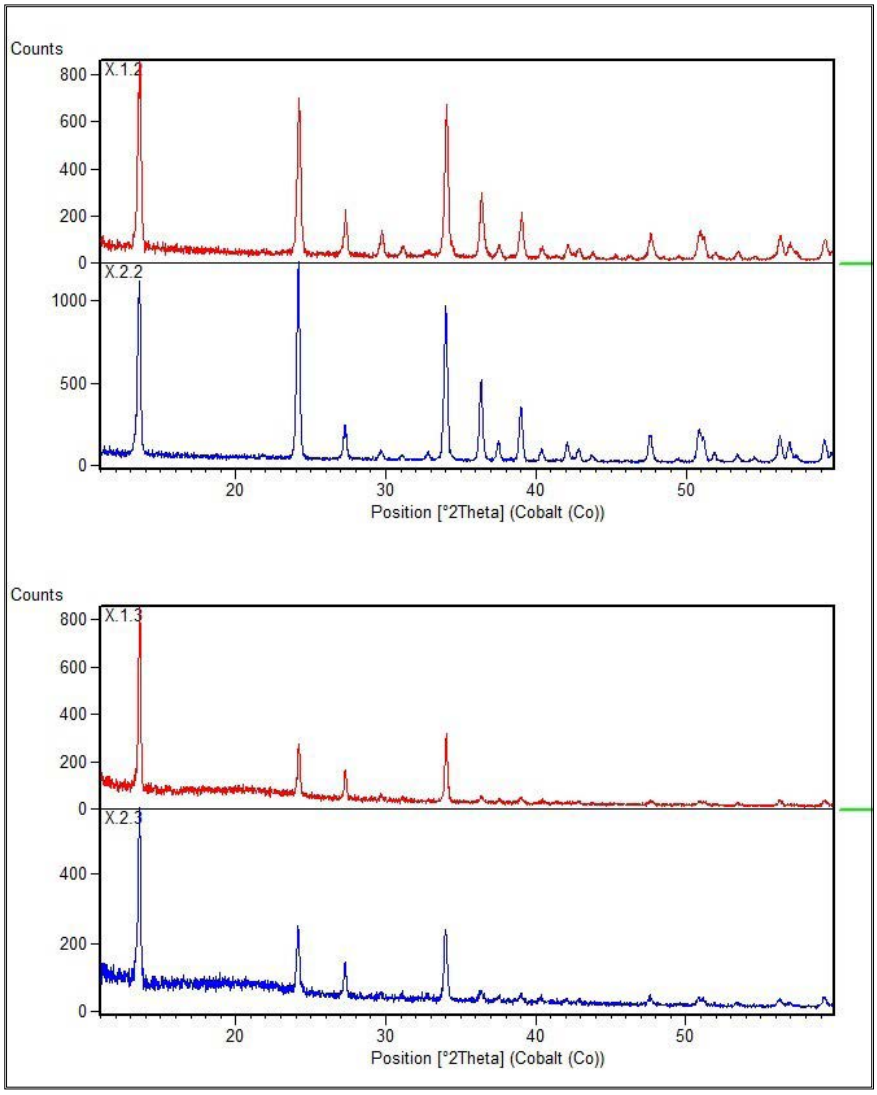


Fig. 6. Top: Comparative phase diagrams of two red color samples, Bottom: Comparative diagram of the phases of the two layers of plaster bed (Authors, 2019). ▶

Conclusion

Examination of the arrays of layers during the two Safavid eras in Qazvin Chehelston monument shows the same execution techniques and materials used.

Both periods of this ornamentation, depending on the minerals used, include gypsum substrate, red soil layer and gold sheets. The differences between the layers of these two periods are also quite minor, depending on the purity of the gold and plaster sheets used in the bed.

In the first period, copper (Cu), zinc (Zn) and slightly tin (Sn) metal impurities are present in the gold layer (X.1.1), which is absent in the second period of the gold layer decorations (X.2.1).

This may be related to the political and economic conditions of governments. This difference can be traced back to extensive studies

of the quality of materials used in different historical periods and to compare them together.

In the base layer and the first period bed (X.1.3), anhydrite mineralization with gypsum was detected which is not seen in the second period bedding (X.2.3).

Investigating this issue requires a case study of its cause. This change reflects the high temperature of the gypsum baking or presence of salts.

The results of this study can be compared with other similar studies on Safavid murals.

The hypothesis of this study is also confirmed that the paintings of the two Safavid periods in Qazvin Chehelston monument are the same in terms of technique of execution according to the near time periods. These paintings can only be different in style. However, this requires further research.

In general, the process of human handicrafts changes when supply does not meet the political, economic, social and cultural needs.

These changes usually take place over a long period of time and are achieved in short periods of time by revolutionary transformations such as warfare or the discovery and development of more efficient techniques.

As a result, two different periods of Safavid paintings on the Qazvin Chehelston monument took place on the same path and for a short period of time and could reasonably have the same principles.

The existence of the second layer in the building can be primarily attributed to the renovation and reconstruction of the building, which still retains its importance and use in the second period. Secondly, this may have been due to the adaption of decorations with the current art style in the new era.

On the other hand, because of the importance of gold in human societies throughout history, the purity of the gold sheets used in the second period may indicate a stabilized and better political and economic situation than the previous period.

The result of this research can be studied more thoroughly by further investigations on murals belonging to different eras of Qazvin Chehelston monument as well as decorations to the rest of the related monuments.

Finally, it is necessary to explain that in this research, the study of organic materials used as fasteners for gold sheets has not been performed. This study is recommended for future research.

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