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Analysis of the Structure of Gray and Azure Glazes of Persepolis Brick, Using XRD and SEM Laboratory Methods

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Abstract: One method of decorating palaces during the Achaemenid period was the use of glazed colored bricks. These glazes encompass a wide range of organic and mineral compounds. This research, conducted in an experimental laboratory, aimed to understand the constituent elements of the glazes on the Persepolis bricks. The main question addressed was which elements are the major determinants of the gray and azure glaze colors on Achaemenid bricks at Persepolis? Among various methods used for mineral identification, XRD analysis was employed to identify materials and determine the phases present, while SEM was utilized to determine the elemental composition. This study focused on the structure and identification of two types of glazes: gray and azure. Samples were physically extracted with a very thin layer of glaze from the sample body. Based on the results obtained, the siliceous and porous nature of the body was confirmed, with iron and magnesium present in the glaze composition, likely part of the primary mix, contributing impurities that affected their coloration. It was also revealed that both gray and azure glazes have an alkaline structure. The findings of this study are crucial for the preservation of ancient artifacts and can guide future research in the field of historical materials.

Keywords: Persepolis, Achaemenid Period, Glazed Bricks, Analysis, XRD method, SEM method.

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Introduction

The use of glazed bricks for decorating palaces and temples dates back to ancient Mesopotamian civilizations. Around 2500 BC, the Sumerians and Akkadians laid the groundwork for classical art in the Near East. The Sumerians used unbaked clay in black, white, and red to cover their walls, while the Babylonians advanced the technique by creating prominent patterns with glazed bricks. In Babylon, large areas of multicolored glazed bricks were used to form parts of structures when arranged side by side (Moortgat and Filson, 1969, 297). During the second millennium BC, the Elamites also employed glazed bricks in their architectural decorations, with the Ziggurat of Chogha Zanbil serving as a notable example (Ghrishman, 1992, 130). In the Achaemenid period, the decoration of palaces and temples reached new heights with extensive use of colorful tiles and painted plaster. Numerous examples of such decorations have been discovered in buildings from this era. For instance, the Immortal soldier reliefs from the Achaemenid palace at Susa exhibit semi-relief painting techniques, where detailed fabric patterns are recognizable (Tajvidi, 1976, 23). Other Achaemenid decorations have been found in places like Pasargadae and Persepolis, including the Treasury at Persepolis. Persepolis, an architectural marvel initiated by Darius the Great in 518 BC, continued to develop until 330 BC when it was destroyed by Alexander the Great, marking the end of Achaemenid rule. Between 1931 and 1935, excavations led by Professor Ernst Herzfeld and architect Professor Crafter, followed by Eric Schmidt and a team from the Oriental Institute of the University of Chicago, uncovered valuable glazed bricks adorned with plant, geometric, and inscriptional motifs. The glazed bricks discovered by Schmidt were distributed to various museums, including the Ancient Iran Museum and the Persepolis Museum. Some bricks are displayed on the eastern wall of the Women's Palace from Xerxes' time, now serving as an administrative building for Marvdasht's cultural heritage. This research focuses on samples located in this administrative building. Due to exposure to environmental and climatic factors, these works are susceptible to damage, making the identification of their constituent elements essential for preservation and maintenance.

Extensive research has been conducted on Persepolis, resulting in numerous books, articles, and investigations. Ernst Herzfeld's work included references to paintings on discovered pottery and glazed bricks in Persepolis palaces (Hertzfeld, 1923; 1927; 1928- 1929-1930; 1933; 1935). Eric Schmidt, responsible for Persepolis excavations from 1935 to 1939, published a three-volume book focusing on the glazed bricks found during his excavations (Schmidt, 1953). Alireza Shapour Shahbazi's "Illustrated Description of Persepolis" introduces the structure of Persepolis and discusses its glazed bricks (Shapour Shahbazi, 1975).

Youssef Nezhad's article studied Achaemenid glazed bricks of Persepolis using various analytical methods, comparing the results with samples from Susa Palace and Elamite glazed bricks from Chogha Zanbil (Yousef Nejad, 2014). Aloiz and Douglas examined gypsum plasters and glazed bricks at Persepolis and Pasargadae, revealing that Persepolis brick pieces are made of silica materials decorated with alkaline glazes (Aloiz/Douglas and Nagel, 2016). Tilia's research on conservation efforts and restorations in Fars included studying Persepolis restorations (Tilia, 1978). Stodulski and colleagues used various analytical techniques to study surface pigments of limestone reliefs at Persepolis (Stodulski/ Farrell and Newman, 2013). Holakooei and colleagues investigated opacifiers and coloring agents in Persepolis glazed bricks, suggesting potential links between Achaemenid glaze industry and Manni glaze production (Holakooei et al, 2016). Emami and colleagues examined the structure of bricks from Tol-e Ajori using XRD and XRF analysis (Emami et al, 2013).

Despite extensive research, no studies have specifically analyzed the constituent elements of the gray and azure glazes of Persepolis glazed bricks. The bricks studied have a completely siliceous

body and are decorated with various glazes, including gray and azure. The severe deterioration of the glaze on these bricks complicates their technical and aesthetic analysis, posing significant challenges in their preservation and display. Understanding the preparation of these brick bodies and glazes, and evaluating optimal preservation conditions, are crucial objectives of this research. Additionally, the study aims to identify the primary elements determining the color of the gray and azure glazes on Achaemenid bricks at Persepolis. This question-oriented research seeks to answer: What are the main constituent elements determining the color of gray and azure glazes of Achaemenid glazed bricks at Persepolis?

Methodology

Various methods exist for identifying materials, with the choice of method depending on the type of materials involved. In the present study, since the focus is on understanding colors, efforts have been made to primarily utilize methods suitable for identifying and analyzing material compositions. This is because this research aims to understand the constituent elements of glazes on Achaemenid glazed bricks at Persepolis. This study is of an experimental-laboratory nature, relying on credible scientific sources and conducted through library research, field studies, and laboratory experiments. In this regard, based on credible scientific sources and conducting library research, along with visits to the historical site of Persepolis and sampling of very tiny layers of glaze from the surface physically and very precisely isolating the paint layers from the glazed bricks installed on the eastern wall of the women's exclusive palace during the time of Xerxes, and performing laboratory methods such as XRD and SEM.

SEM analysis was carried out in the central laboratory of Isfahan University of Technology using a Philips XL30 Scanning Electron Microscope (SEM) made in the Netherlands. Initially, the samples were taken from the surface of the bricks of Persepolis, thoroughly washed with acetone, and finally powdered. The powdered samples were placed on carbon tape and then coated with a layer of gold (a conductive material). The thickness of this layer, which prevents surface charging and preserves the surface characteristics of the sample, is 10 nanometers. The samples were then placed in the device's sample chamber, and the test was performed. XRD analysis was carried out in the central laboratory of Isfahan University of Technology using a Panalytical X'Pert X-Ray Diffraction (XRD) device made in the Netherlands. Initially, the samples were taken from the surface of the bricks of Persepolis, thoroughly washed with acetone, and finally powdered. About 0.01 grams were placed in the device chamber, and X-ray diffraction was performed. In this system, the tube and sample holder are movable, and the detector is fixed, with a 2θ angle ranging from 5 to 70 degrees ($2\theta = 5-70$) with step increments of 0.02 degrees and a dwell time of 1 second per step.

Sample Description

Glazed bricks from various palaces at Persepolis were discovered during Eric Schmidt's excavation, and then installed on the eastern wall of the women's exclusive palace during the Xerxes, which is currently used as the administrative building of the cultural heritage of Marvdasht. It is presumed that they were also installed in the same manner during the Achaemenid era (*Figure 1*). The appearance of these bricks is very porous, resembling shiny milk-colored stone. They are glazed with white, light green, dark green, dark brown, fawn yellow, and gray colors, with designs separated by azure lines.

Considering that the appearance of the body is similar to small granular particles with sharp corners stuck together (like sharp-tipped sand grains stuck together with a lot of empty space between them), it is likely that very fine silica particles mixed with some lime have been baked, with lime

acting as a flux aiding in the fusion of silica particles. It is also possible that the body composition existed as a ready-made mixture in nature and was baked without adding other materials. On the other hand, the body may have been baked at a temperature where 16% calcium oxide for a composition with a high silica content was insufficient as a flux, necessitating a very high temperature unless at a low temperature, additional materials were added as fluxes during firing, ultimately blending together during firing and undergoing phase changes.

The glazed bricks under study, due to being exposed to outdoor environments for many years (temperature, humidity, light, atmospheric pollutants), have been constantly undergoing degradation and physical and chemical changes. Therefore, it is observed that the surface of the glazes is very rough, uneven, and opaque. The glazed bricks under study have relatively similar dimensions, all being 9 centimeters in height and 35 centimeters in length, with variable widths ranging between 10 to 20 centimeters. These bricks are almost similar to square tiles used in dome coverings, with the back of the bricks narrower than the front, measuring 6 centimeters. Additionally, on the side of the bricks that are not glazed, small colored glaze spots have been marked, which are likely the signature of the piece's maker (Hertzfeld, 2002, 243).







Figure 1: Layout of the investigated bricks

Discussion

To conduct the necessary experiments, samples of the glazes under study needed to be transferred to the central laboratory of Isfahan University of Technology. However, due to the installation of the glazed bricks on the eastern wall of the women's exclusive palace during the time of Xerxes, it was not feasible to transport them to the laboratory. Therefore, sampling was carried out. During sampling,

due to the very tiny flaky layers of glaze physically separating from the body and the impossibility of precise separation of the color layer from the glazed bricks installed, a portion of the body along with the glaze was transferred to the laboratory. During the experiment, in order to prepare the samples as powders, it is possible that due to the siliceous nature of the body, a percentage of the silica identified in the glazes was related to the body beneath the glaze (*Table 1*).

Table 1: Adjusting the glaze of the studied samples with MUNSELL's table (Authors)

No.	Samples	Glaze samples	Number	Glaze color and its code based on MUNSELL table	Adjusted colors of MUNSELL table
1	Gray color		3	10bg 4/2	
2	Azure color		3	5Pb 2/8	

Gray Glaze Oxides

“Iron oxide in glazes, in an oxidizing firing environment, first changes the color to yellow, then to reddish-brown, wine-red, and finally to brown. This oxide, in a reducing firing environment, produces other colors such as bluish-gray to dark gray. When iron oxide saturates the glaze, unlike manganese and copper compounds, metallic appearance is not evident on its surface, but only the surface of the glaze is matte” (Abbasian, 1991, 115). *Table 2* and *Figures 2* and *3* illustrate the test results on gray glaze. *Table 2* pertains to the gray color, showing a main amorphous phase and a quartz phase, the appearance of the gray glaze layer is a completely matte color, indicating gray with a greenish hue.

Based on the XRD test results and examination of SEM *Figure 2* and considering the explanations provided regarding coloring oxides for gray, it is possible to speculate about this sample. In the SEM diagram belonging to the gray glaze, the presence of elements aluminum (Al), potassium (K), calcium (Ca), iron (Fe), and sodium (Na) is among the most prominent elements, likely originating from aluminum oxide (Al_2O_3), potassium chloride (KCl), calcium oxide (CaO), iron oxide (Fe_2O_3), and sodium oxide (Na_2O). Additionally, the presence of chromium (Cr) and titanium (Ti), originating from chromium oxide (Cr_2O_3) and titanium dioxide (TiO_2), should not be overlooked.

The presence of potassium (K), sodium (Na), calcium (Ca), and the absence of lead (Pb) indicate that the gray glaze is solely an alkaline glaze. Copper (Cu), chromium (Cr), and iron (Fe) are coloring agents, with copper (Cu) and chromium (Cr) in this (alkaline) glaze producing a turquoise color, while iron (Fe) under reducing conditions produces a gray color, as observed in SEM diagram number 2, where the presence of iron (Fe) is more significant and preferred over copper (Cu) and chromium (Cr). Although the presence of titanium (Ti) may not be very pronounced, even this amount can significantly affect the matte appearance of the gray color. The presence of titanium oxide (TiO_2) in the glaze may have been unintentional, although many other oxides may have unintentionally been present in the glaze composition at that time.

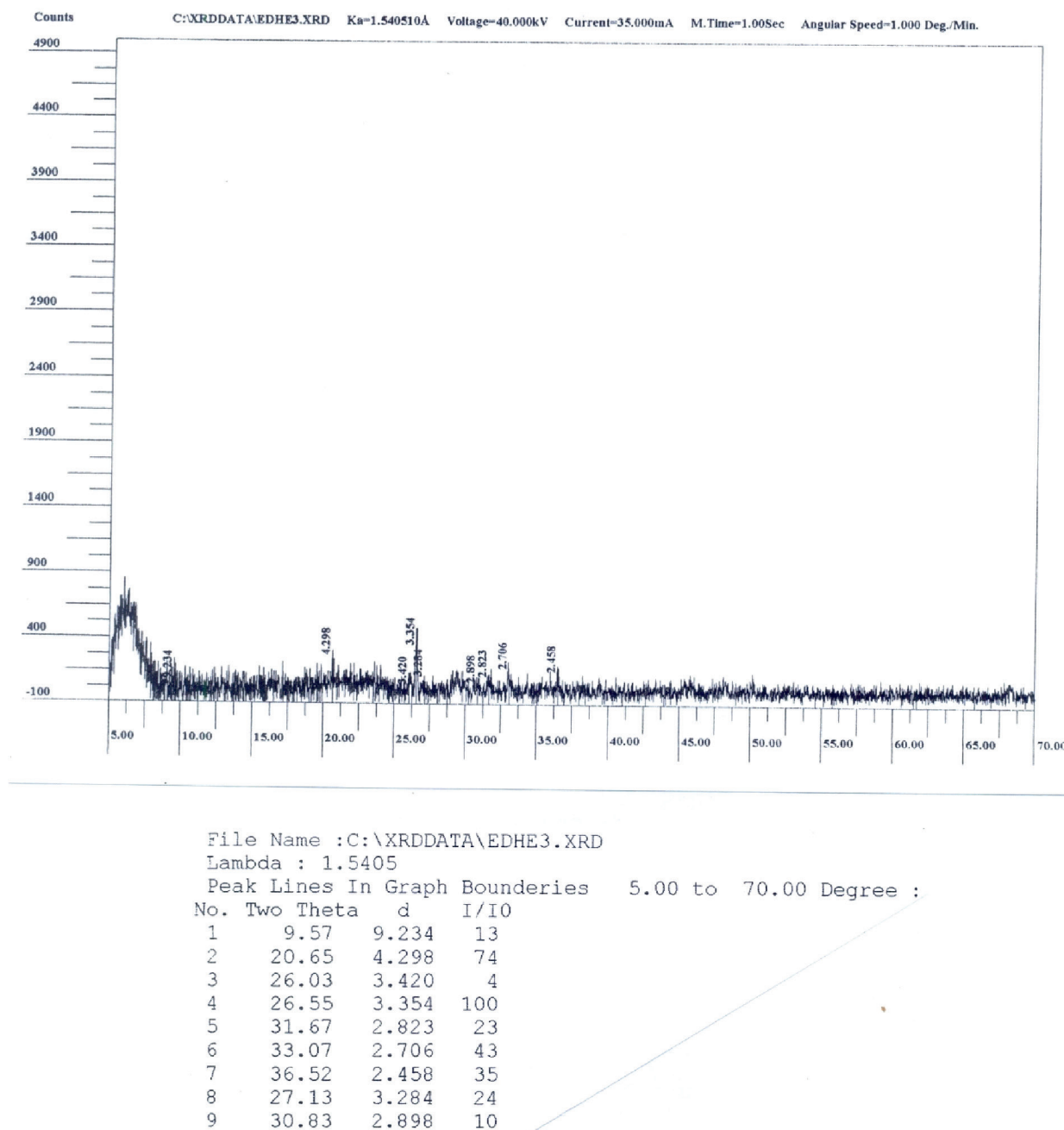


Figure 2: XRD test of gray color related to the gray XRD graph

“Titanium oxide (TiO₂) imparts a yellow color to lead glazes and a white color to lead-free glazes. However, with the presence of a small amount of iron oxide (Fe₂O₃) in the glaze or in titanium oxide (TiO₂), it turns yellow again. The presence of titanium in large amounts in the glaze makes it matte” (Abbasian, 1991, 116). Finally, the alkaline nature of this glaze (sample 1) and the likely main factor in the gray color being the combined presence of iron (Fe), calcium (Ca), and titanium (Ti) (from oxides of Fe₂O₃, CaO, TiO₂) make this glaze less transparent compared to other glazes. The presence of gold (Au) in SEM **Figure 3** is not related to gold in the glaze and color but is related to the electron microscopy system since electron rays hit the sample under the electron microscope, a 20-nanometer-thick layer of gold coating is applied to the sample for thermal electrical conduction, causing the gold peaks seen in the SEM diagram.

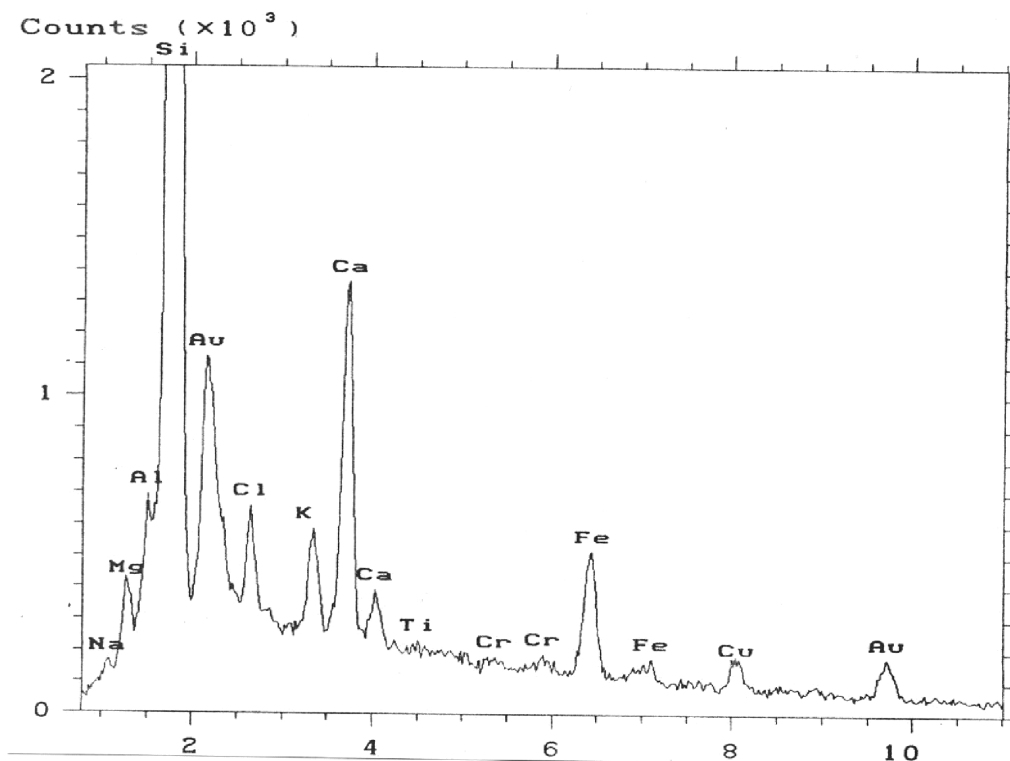


Figure 3: The result of the SEM test of gray color

Figure 4 shows a microscopic image of the surface of the gray glaze, magnified 2700 times. This microscopic image of the gray glaze surface indicates the color due to the fact that the phases present in the color are not well separated from each other, and the boundary between the crystalline grains is

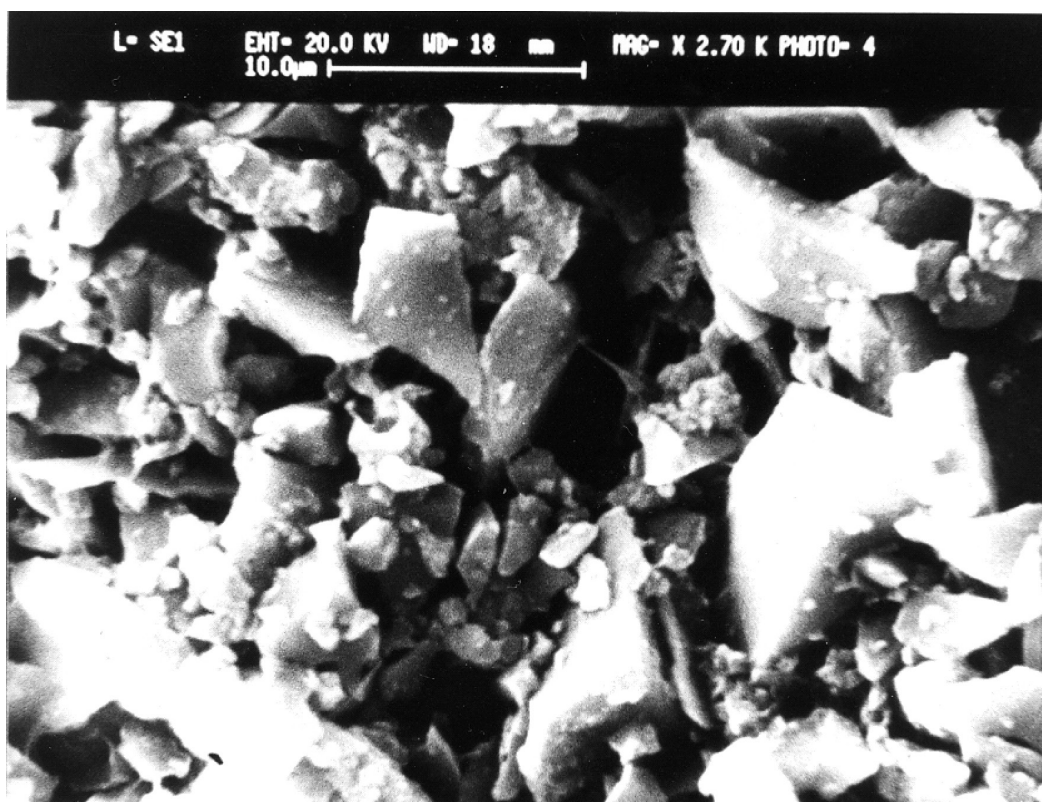


Figure 4: Microscopic image of gray glaze

not clearly defined, almost showing a single phase. Therefore, this image represents the surface of the sample and does not show the shapes of the phases and crystalline particles.

Azure Glaze Oxides

With a gradual increase in the amount of cobalt compounds (CoO) in the glaze, light blue to dark blue colors emerge. Usually, various blue colors consist of a mixture of cobalt compounds (CoO) with aluminum oxide (Al₂O₃) and even some zinc oxide (ZnO). Cobalt compounds (CoO) with phosphates and arsenates produce blue-purple to dark purple colors, which become more intense with the addition of magnesium oxide (MgO) (Sabia, 1999, 123). “Replacing flux materials with lead oxide (PbO) in the glaze tends to shift the glaze color towards blue-green, and upon reaching complete alkalinity, its color becomes entirely blue” (Barsoum, 1997, 199).

Azure glazes, after sampling, appeared as flaky layers. It is necessary to mention that due to the impossibility of precise separation of the color layer from the glazed bricks installed in Persepolis, a percentage of the substrate under the color was accompanied by glaze, and due to the siliceous nature of the substrate, a percentage of the silicon identified in the glazes was probably related to the substrate. Azure glaze has been penciled around the designs on glazed bricks, and other colors on the bricks have been separated from each other by these lines.

The blue color of azure glaze can only be produced by a mixture of this oxide and a strong alkaline lead-free glaze. If borax acid is added to a green glaze, the color of this glaze becomes turquoise, and by adding 8 to 10 percent of lead to the same glaze, the intensity of turquoise color appears more prominent (Pampuch, 1976, 78). XRD and SEM tests were performed on samples of azure glaze, and the results obtained are shown in *Table 3* and *Figures 5* and *6*.

Table 4 and *Figure 5* show the presence of two phases, quartz SiO₂ and “Hedon Berylite” Ca (Fe, Mn) Si₂O₆, in the tested samples of azure glaze. In addition to the XRD test results on azure glazes and considering *Figure 6*, which is the result of SEM testing on this type of glaze, it shows that copper in alkaline glazes creates a blue color, while in lead glazes, it creates a green color. In this diagram, apart from silica, which is the main substance of this type of glaze, Fe comes from iron oxide (Fe₂O₃), Ca from calcium oxide (CaO), K from potassium chloride (KCl), and Cu from copper oxide (CuO).

Table 2: XRD test results of gray and azure color (Authors)

Test No.	Sample No.	Result
EDHE3	Gray	Amorphous phase (main) + low Content of Quartz. SiO ₂ , 33-1161
EDHE1	azure	Quartz, SiO ₂ 33-1161, main PPhase + Ca (Fe,Mn)Si ₂ O ₆ 41-1372 Hedenbergite

Initially, the presence of cobalt oxide (CoO) in the composition of the azure glaze is assumed upon observing the glaze. However, the examination of the SEM test result, depicted in *Figure 6*, indicates the absence of cobalt oxide (CoO) in the azure glaze. Since this glaze (sample 2) was applied on a piece of glazed brick directly placed under the SEM device, the likelihood of error in the experimental sampling is very low. Moreover, this diagram (*Figure 6*) indicates the absence of lead (Pb), hence the above glaze is an alkaline glaze. It has been previously stated that in lead glazes, by substituting an alkaline agent instead of lead oxide (PbO) to the extent of complete alkalinity, the color changes from green to blue-green and then entirely to blue. It is possible that at that time, by substituting an alkaline agent instead of a lead-based agent (in lead glazes), they obtained the azure glaze, and essentially, at

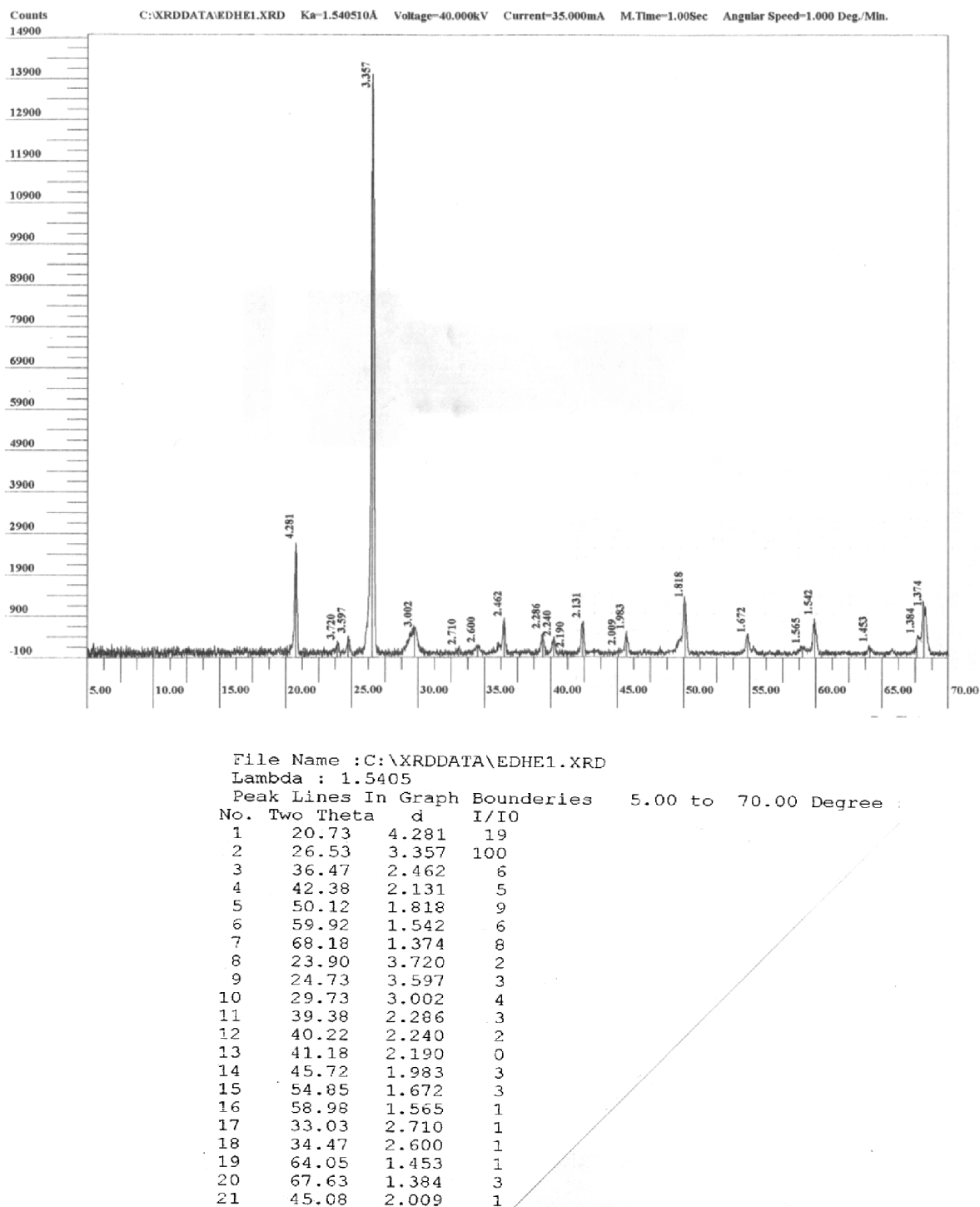


Figure 5: Azure XRD test related to the azure XRD graph

that time, by adding a raw material containing alkaline substances instead of a raw material containing lead (such as ceruse, etc.), they underwent a color transformation (from green to blue).

The presence of iron (Fe) in SEM **Figure 6** cannot be indicative of the blue colorant in the above glaze composition. However, if cobalt oxide (CoO) is proven to be present in the above sample (azure glaze), the blue color of this glaze was undoubtedly influenced by cobalt, even though copper (Cu) is also present in the composition. **Figure 7** shows a SEM microscopic image of the azure glaze.

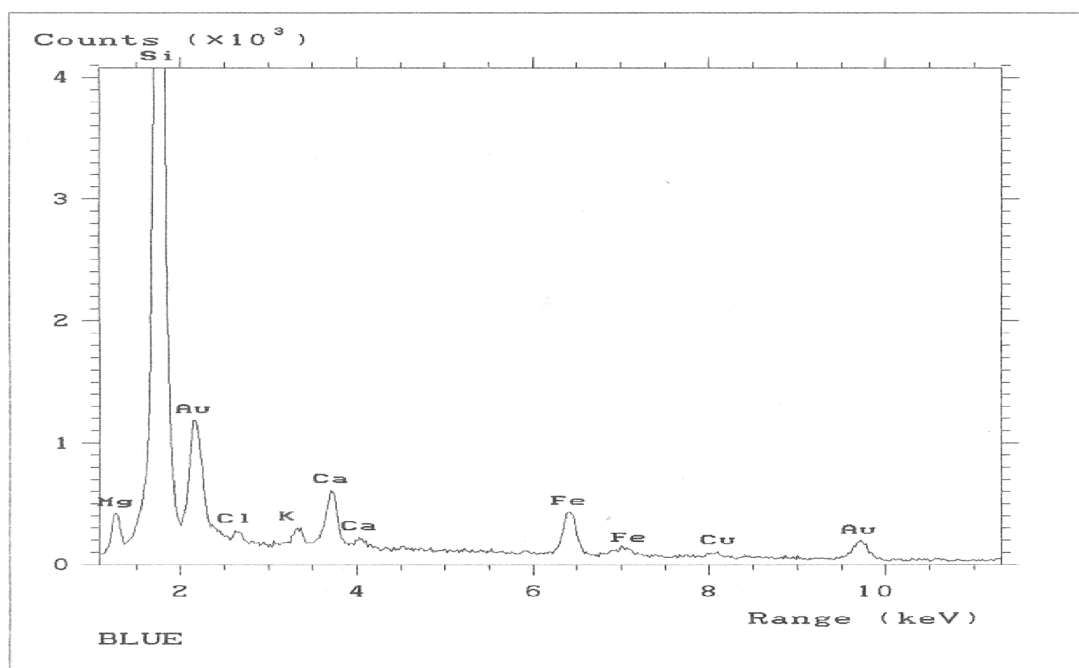


Figure 6: SEM test result of azure color

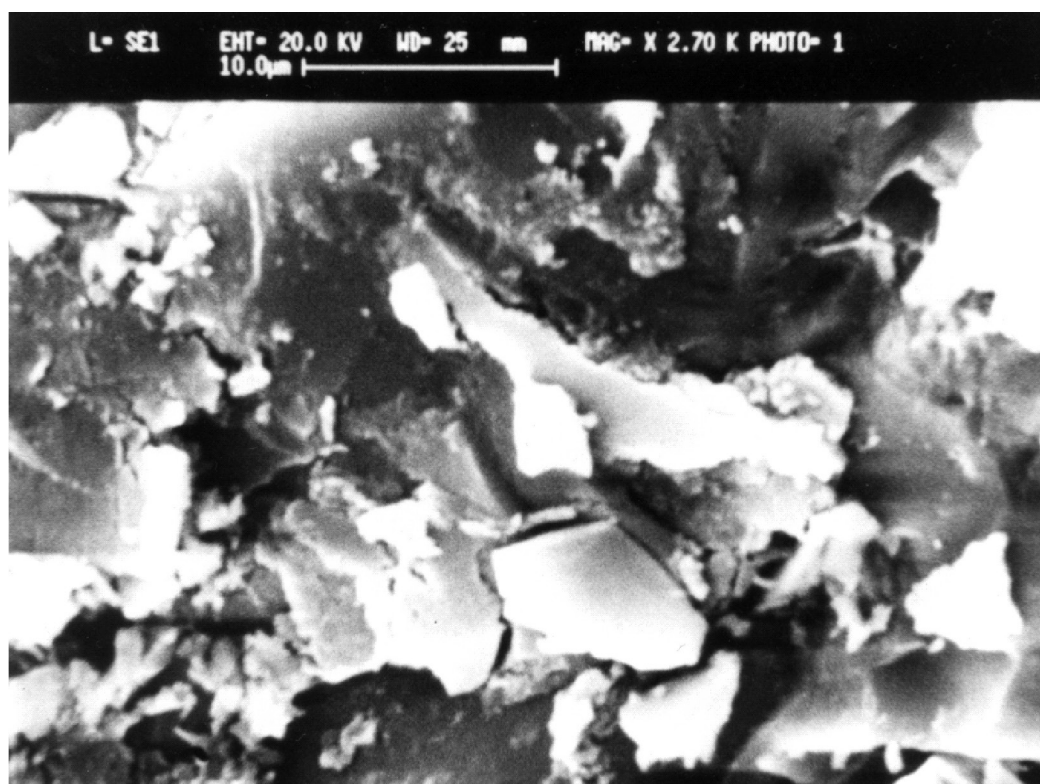


Figure 7: Microscopic image of azure glaze

Considering the alkalinity of the azure glaze and copper oxide (CuO) as the coloring agent, a formula similar to the tested azure glazes can be obtained using the formula in *Table 4*. Additionally, 3% copper oxide (CuO) should be added to the above composition. This glaze is suitable for SK03a, which means a temperature of 1040 degrees Celsius. If we assume a lead glaze and also consider cobalt as the coloring agent, we can use the formula in *Table 5*.

Table 4 The formula of an azure glaze (Authors)

Silica SiO_2	1.7	Aluminum oxide Al_2O_3	0.05	Sodium oxide Na_2O	0.3
				Potassium oxide K_2O	0.7

Table 5: Lead glaze formula with cobalt coloring agent (Authors)

Quartz SiO_2	1.7	Aluminum oxide Al_2O_3	0.17	Lead oxide PbO	0.55
		Boric acid B_2O_3	0.1	Zinc oxide ZnO	0.15
				Calcium oxide CaO	0.10
				Barium oxide BaO	0.10
				Lithium oxide Li_2O	0.10

Based on the analysis of the test results, quartz was identified as the main phase forming the bricks, which are uniformly distributed in the brick structure. The glaze used on the bricks is based on alkaline compounds. Additionally, considering the severe erosion of the glazes, which now manifest themselves with color fading and numerous pits on the glaze surface, the recognition of the historical structure of the glazes is very difficult yet crucially important. Overall, based on laboratory studies and structural analysis of Achaemenid glazed bricks in Persepolis, despite the high impurities such as iron oxide and calcium oxide in the marl soil, these bricks can be classified as relatively high-quality. The process of making all samples, both in terms of processing and raw material preparation, as well as in terms of baking techniques, has been uniform. This uniformity can indicate attention to the selection of raw materials and predetermined stages for baking the bricks. Regarding the glazes, experiments focused on identifying the constituent elements of the glazes and examining the surface condition in terms of erosion and weathering processes. For this purpose, SEM and XRD methods complemented each other, providing the desired information about the glazes, while also confirming and identifying the constituent elements of the glazes, which had not been done in this way before.

Based on the discoveries made by archaeologists and the collection of remaining artifacts from the Achaemenid period, a significant portion of which includes unglazed bricks, glazed bricks, and glazed embossed bricks, studies on these artifacts from various technical and artistic aspects will be of interest to archaeology and the restoration of artifacts. Previous research and studies have mostly focused on the type of raw material and the method of preparation and production of brick bodies while identifying the glaze composition on the bricks has not been given much attention. This research addresses this aspect. The similarity in the identified elements and phases indicates that the artists, craftsmen, and makers of Achaemenid glazed bricks followed a series of basic standards in terms of raw materials and firing techniques during the manufacturing process. This can indicate the expansion of Achaemenid industries and trade networks.

Conclusion

Based on the conducted studies, the appearance of the glazed bricks found in Persepolis appears to be very porous, with a milky color similar to shiny limestone. The siliceous nature of the bricks has been confirmed. Quartz was identified as the main phase of the bricks, and the observation of quartz grains with angular corners suggests that these materials were used as fillers to fill the voids created by the erosion of the parent rocks. The quartz grains are uniformly dispersed in the brick texture, indicating good mixing of these filler materials. Gray and azure glazes used on the glazed bricks have been alkaline, which may also be the reason for the matte appearance of these glazes, in addition to the effects of time and environmental factors. In the composition of all glazes, iron and magnesium

were present, which, apart from their color effects in the main composition of the base glaze, also had an impurity effect. XRD analysis of gray glaze shows a main amorphous phase and a quartz phase, and the appearance of the gray glaze layer is completely matte, with a gray color showing a hint of green tint. SEM analysis of gray glaze shows the presence of aluminum (Al), potassium (K), calcium (Ca), iron (Fe), and sodium (Na), which are likely from aluminum oxide (Al_2O_3), potassium chloride (KCl), calcium oxide (CaO), iron oxide (Fe_2O_3), and sodium oxide (Na_2O), respectively. It should be noted that the presence of chromium (Cr) and titanium (Ti), which are from chromium oxide (Cr_2O_3) and titanium dioxide (TiO_2), should not be overlooked. The presence of potassium (K), sodium (Na), calcium (Ca), and the absence of lead (Pb) indicate that the gray glaze is purely alkaline. XRD analysis of lapis lazuli glaze shows the presence of two phases: quartz SiO_2 and “Heden Beryllite” $\text{Ca}(\text{Fe}, \text{Mn})\text{Si}_2\text{O}_6$. SEM analysis of azure glaze reveals the presence of iron (Fe) from iron oxide (Fe_2O_3), calcium (Ca) from calcium oxide (CaO), potassium (K) from potassium chloride (KCl), and copper (Cu) from copper oxide (CuO), in addition to silicon (Si), which is the main component of the glaze. The limitations of the study include the difficulty in collecting samples due to the fragile and deteriorated state of the glazes, which led to some of the underlying body material being included in the samples. Additionally, the precise separation of the glaze from the body was not always possible, potentially contaminating the glaze samples with silica from the substrate. These factors complicate the analysis and identification of the glaze’s constituent elements.

The results of this research have applications in the conservation and restoration of Achaemenid glazed bricks in Persepolis and the education of students in the preservation of cultural and historical artifacts at various levels. Furthermore, it will be highly effective in conservation and restoration policy-making for the Persepolis complex. In this regard, future research could focus on topics such as identifying the constituent elements of other glazes on Achaemenid bricks at Persepolis, as well as identifying the constituent elements of the glazes on Achaemenid bricks in other structures from this period.

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