



## Archaeometric Review of the Tile Decorations in Hasankeyf Zeynel Bey Tomb with P-EDXRF

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Tile is known as a handicraft based on the production of items such as vases, jugs and pots by shaping the soil after cooking. It is defined as the covering material processed with colorful décor and motifs used in the decoration of items such as tiles, ceramics, porcelain plates and china making in the goods processed with the help of this material.

In this study, 7 Light Blue Glaze, 7 Navy Blue Glaze and 8 Unglazed Tile belonging to Zeynel Bey Tomb, a total of 22 tiles were analyzed in Hasankeyf Cultural Park where the tomb was moved with P-EDXRF.

As a result; The chemical contents of the 22 samples we examined on site were determined and compared with the previous studies. It was determined that the results obtained were compatible.

## Hasankeyf Zeynel Bey Türbesi'ndeki Çini Bezemelerinin P-EDXRF ile Arkeometrik İncelemesi

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Çini toprağın pişirildikten sonra şekil verilerek vazo, sürahi, kap-kacak gibi eşyaların üretilmesine dayalı el sanatı olarak bilinmektedir. Fayans, seramik, porselen tabak gibi eşyaların süslenmesinde kullanılan renkli dekor ve motiflerle işlenmiş kaplama malzemesi ve bu malzeme yardımı ile işlenmiş eşyalarda çinicilik olarak tanımlanmaktadır.

Bu çalışmada, Zeynel Bey Türbesine ait 7 adet Açık Mavi Sır, 7 adet Lacivert Sır ve 8 adet Sırsız Kiremit olmak üzere toplamda 22 adet Çini'nin P-EDXRF ile Türbenin taşındığı Hasankeyf Kültürel Parkta yerinde analiz yapılmıştır. Sonuç olarak; yerinde incelediğimiz 22 adet numunenin kimyasal içerikleri belirlenip daha önce yapılmış olan çalışmalar ile karşılaştırılmıştır. Elde edilen sonuçların uyumlu olduğu tespit edilmiştir.

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## 1. INTRODUCTION

Hasankeyf, whose history dates back to ancient times, is located in the enterprise region of Northern Mesopotamia and Anatolia. Its establishment as a settlement dates back to the present day from prehistoric times. It was used as an important military base during the Byzantine and Romans period.

The Zeynel Bey Tomb in the Hasankeyf district of Batman was located in the northern part of the Tigris River, west of the Hasankeyf bridge, before it was moved to the New Cultural Park area. It is known that the tomb was built for Zeynel Bey, the son of Uzun Hasan, the ruler of Akkoyunlu (Yurttaş, 1996). The fact that there is no other example of Zeynel Bey's tomb in Anatolia increases its importance even more. The architect of the tomb was Pîr Hüseyin, son of Abdurrahman of Iranian origin (Gabriel, 1940). It is thought that the monument can only be a master of tiles, but also as a builder. The cylindrical body, which rises on a circular substructure without moving, is standing but in a dilapidated state. The lower part of the hull was cast stone coverings, the upper part of the hull and the outer surface of the dome were destroyed over time. The interior of the building also suffered from ruins, the cover of the burial chamber was destroyed, the covering tile plates were destroyed, and there is no trace of the tomb (Citizen, 1996).

It is a product obtained by shaping the dough formed by mixing a certain ratio from raw materials such as tile, chalk, clay, kaolin and quartz, priming, first cooking and glazing, and finally the second cooking (Atalay, 1983).

The raw materials used in the making of tile are in two forms, glazed and unglazed tile raw materials. When kneaded with water, they do not disperse, have a structure suitable for shaping and at the same time retain their shape when dried; Substances such as clay, kaolin and bentonite are known as cored tile raw materials (Arcasoy, 1983). Glazed Tile Raw Materials, on the other hand, although they are finely ground, do not take shape easily with water. Even if they are shaped, they lose their shape and disperse in case of any external influence. Quartz and Dolomite (chalk) are known as raw materials of unglazed tile samples (Arcasoy, 1983).

Zeynel Bey Tomb was moved 2 kilometers with the project implemented for the first time in Turkey on May 12, 2017 and it was removed from possible dam lake area. The world's most advanced technologies were used in the transportation of the Zeynel Bey Tomb, which weighed 1100 tons. The tomb was placed in its new location, Hasankeyf New Cultural Park Area, with 8 "Self-Propelled Modular Carriers" special platforms with 192 wheels, after a successful work of about 4 hours (Figure 1).

According to our source research, there is only one study with the analysis of the materials used in the construction of the Zeynel Bey Tomb. In the study conducted by Eskici and et al. (2008), they examined the structure of the materials used in the construction of the tomb.

Archaeometry is formed by the combination of the words Archaeology and metric and is known as the system and measurement used in the evaluation of qualitative and quantitative analyzes of archaeological remains (Taylor and Aitken 1985). In recent years, archaeometry studies, in which historical artifacts and finds are analytically examined, have attracted the intense attention of scientists (Aydın 2017; Işık 2019; Öztoprak 2019; Avan 2020). Portable-Energy Dispersive X-Ray Fluorescence Spectrometer (P-EDXRF) used in the realization of these analyzes is considered to be an advantage that that it is non destructive, it let us to make insitu analysis, the results can be taken very fast and its usage is simple and easy.

When all this information was evaluated, it was decided to evaluate the results obtained by in situ analysis technique by using P-EDXRF spectrometer to determine the chemical structure of the "Tile Decorations in Hasankeyf Zeynel Bey Tomb" archaeometrically. A total of 22 tiles, including 7 Light Blue Glaze, 7 Navy Blue Glazes and 8 Unglazed Tile belonging to the Tomb of Zeynel Bey, were analyzed with P-EDXRF. Before the construction of the Ilisu dam and the water retention in a large area, the historical artifacts that could possibly be under water in Hasankeyf were moved to the Hasankeyf Cultural Park areas with the latest technologies. The Tomb of Zeynel Bey is among these historical monuments (Figure 1). With this relocation process, the "Zeynel Bey Tomb", especially Hasankeyf, attracted attention. Considering all these, it is extremely important that the Archaeometric

study of the "Tile Decorations in the Tomb of Zeynel Bey" is carried out using the P-EDXRF spectrometer, which offers a non-destructive, practical, economical, simple and fast method.

This archaeometric study on Zeynel Tomb tiles and unglazed brick samples has successfully revealed the chemical composition of the materials. It is inevitable that the P-EDXRF device used in this study will be preferred in cases where destruction is not desired in archaeometric studies. With this method, which provides great convenience in the examination of immovable and undestructive cultural assets, detailed information about the structure and glaze compositions of the materials subject to the study is reached.

## 2. MATERIAL AND METHOD

A total of 22 tiles, including 7 Light Blue Glazes (Figure 2), 7 Navy Blue Glazes (Figure 3) and 8 Unglazed Tile (Figure 4) belonging to the Zeynel Bey Tomb, were analyzed on-site in Hasankeyf Cultural Park, where the tomb was moved with P-EDXRF.

In order to obtain healthier results before the analysis was made on the tiles, the area to be analyzed was analyzed after cleaning the dust, dirt and limestone layer on it with the help of a soft-tipped brush and scalpel.

Analysis was performed with the P-EDXRF (Olympus, Delta Premium) device, which is a non-destructive method to determine which chemical components ( $\text{SiO}_2$ ,  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Cu}$ ,  $\text{Co}$ ,  $\text{Sn}$ ,  $\text{Pb}$ ,  $\text{S}$ , etc.) are in the chemical structure of the tiles. After the appropriate time and mode of the P-EDXRF instrument are set, the instrument is placed on the sample and the analysis begins. In order to achieve a repeatable and reliable result, it is extremely important to hold the device steadily without moving. After the device is held stable for about 2-3 minutes, the relevant analysis results are recorded in % and ppm on the screen. The results are then transferred to the computer environment.



**Figure 1.** A general view from the relocation phase of the Zeynel Bey Tomb. (<https://www.ntv.com.tr/galeri/sanat/tarihi-zeynel-bey-turbesi-tasindi,b0b56QPw9U-2A4yZIXArwQ/p7bCaPmVF0SXv3TlrAic9A>).



**Figure 2.** Analysis of the Light Blue Tiles of Zeynel Bey Tomb with P-EDXRF



**Figure 3.** Analysis of the Navy Blue Tiles of Zeynel Bey Tomb with P-EDXRF



**Figure 4.** Analysis of the Unglazed Tiles of Zeynel Bey Tomb with P-EDXRF.

### 3. RESULT AND DISCUSSION

The results of P-EDXRF analysis of navy blue glaze, light blue glaze and unglazed tiles are averaged and given in figure 5 and Table 1. In light blue glaze samples,  $\text{SiO}_2$  was found to be between 52.94% and 60.31%. The  $\text{SiO}_2$  mean of 7 samples was calculated as 56.82%. The CaO compound is the 2nd most common compound in these samples. The compound was found to be between 3.42% and 5.84%. It has an average of 4.20% in samples. In light blue glaze samples, the  $\text{K}_2\text{O}_5$  compound was between 2.96% and 3.00% and averaged 3.25%. The  $\text{Fe}_2\text{O}_3$  compound was found in these samples at between 1.07% and 1.68%. The average of  $\text{Fe}_2\text{O}_3$  is 1.37%. The  $\text{Al}_2\text{O}_3$  compound averaged between 0.93% and 2.04% in light blue glazed samples, with an average of 1.25%. Pb (12.07% - 14.83%), S (6.03% - 7.34%), Sn (3.50% - 4.47%) and Cu (1.66% - 2.53%) were detected only in open-glazed samples and their mean values were calculated as 13.(Pb) 63%, (S) 6.85%, (Sn) 3.90% and (Cu) 2.17%, respectively.

In navy blue glaze samples,  $\text{SiO}_2$  was found to be between 82.06% and 73.66%. The  $\text{SiO}_2$  mean of the 7 samples was the most abundant compound at 78.32%. The CaO compound is the 2nd most common compound in these samples. The compound was detected in the range of 3.10% to 6.85%. It has an average of 5.13% in samples. In navy blue glaze samples, the  $\text{K}_2\text{O}_5$  compound is between 1.08% and 4.76% and has an average of 3.18%. The  $\text{Fe}_2\text{O}_3$  compound was found in these samples at between 2.82% and 3.75%. The average of  $\text{Fe}_2\text{O}_3$  is 3.42%. The  $\text{Al}_2\text{O}_3$  compound is between 0.83% and 1.54% in navy blue glazed samples and has an average of 1.10%. In the navy blue glaze samples, the element Co was detected between 0.44% and 0.85%. The average value in these samples is 0.68%.

SiO<sub>2</sub> was found to be between 30.88% and 51.61% in unglazed samples. The SiO<sub>2</sub> mean of the 7 samples was the most abundant compound at 41.94%. The CaO compound is the 2nd most common compound in these samples. The compound was detected in the range of 12.08% to 33.13%. It has an average of 18.80% in samples. In unglazed samples, the K<sub>2</sub>O<sub>5</sub> compound is between 1.22% and 2.89% and has an average of 2.09%. The Fe<sub>2</sub>O<sub>3</sub> compound was found in these samples at between 4.96% and 9.29%. The average of Fe<sub>2</sub>O<sub>3</sub> is 7.94%. The Al<sub>2</sub>O<sub>3</sub> compound was between 6.39% and 12.19% in unglazed samples and averaged 9.35%. In unglazed samples, the MgO compound was detected between 2.21% and 6.62%. The average value in these samples is 3.43%.

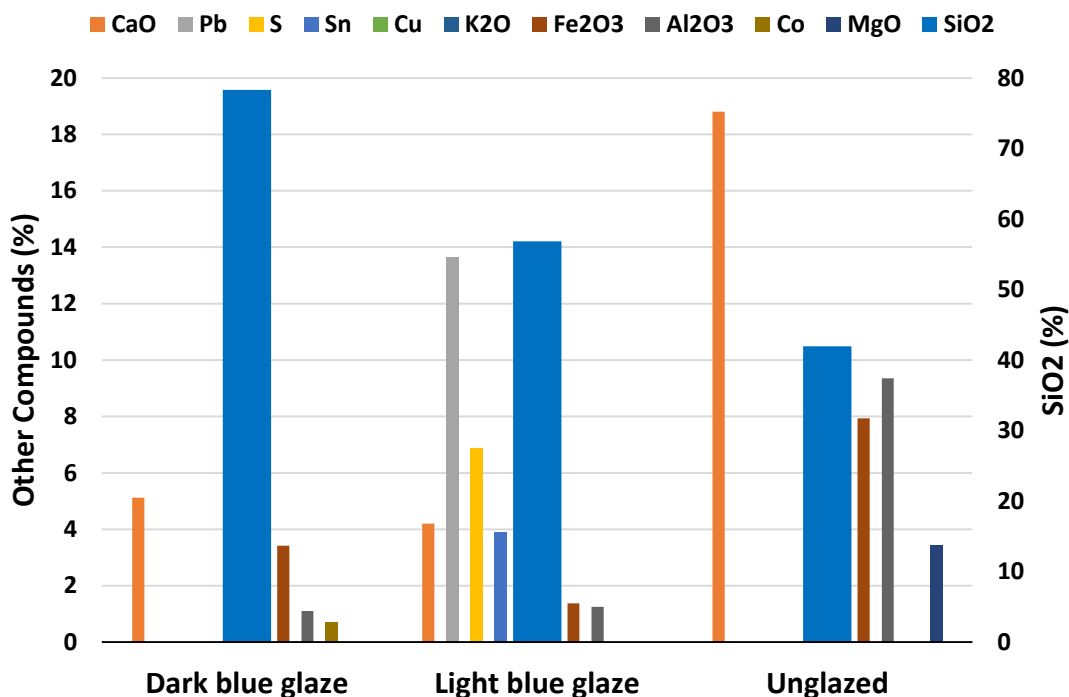


Figure 5. P-EDXRF Analysis Distribution Rates of Hasankeyf Zeynel Bey Tomb Tile Samples.

Table 1. P-EDXRF Analysis Results Average of Hasankeyf Zeynel Bey Tomb Tile Samples.

|                                | Dark blue glaze | Light blue glaze | Unglazed |
|--------------------------------|-----------------|------------------|----------|
| SiO <sub>2</sub>               | 78.32           | 56.82            | 41.94    |
| CaO                            | 5.13            | 4.20             | 18.80    |
| K <sub>2</sub> O <sub>5</sub>  | 3.18            | 3.25             | 2.09     |
| Fe <sub>2</sub> O <sub>3</sub> | 3.42            | 1.37             | 7.94     |
| Al <sub>2</sub> O <sub>3</sub> | 1.10            | 1.25             | 9.35     |
| MgO                            | ND              | ND               | 3.43     |
| Pb                             | ND              | 13.63            | ND       |
| S                              | ND              | 6.85             | ND       |
| Sn                             | ND              | 3.90             | ND       |
| Co                             | 0.68            | ND               | ND       |
| Cu                             | ND              | 2.17             | ND       |

#### 4. CONCLUSION

According to the results of P-EDXRF analysis in this study, SiO<sub>2</sub> compound was determined at a high rate compared to other chemical components in all samples. When Table 1 and Figure 5 were examined in detail, the elements Pb, S, Sn and Cu were detected only in the light blue glazed samples. It can be said that the formation of this glaze color is formed from a mixture of these elements. In addition, the presence of the element Co only in navy blue glazes caused the formation of this glaze color. MgO was only observed in unglazed samples. The basic components of unglazed samples are known as CaO and MgO. This result has been found to be compatible for unglazed samples. In unsorted sample samples, the rate of SiO<sub>2</sub> decreases, while the rate of CaO increases. In addition, it is extremely important that the levels of CaO, Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> compounds are increased in unglazed samples and that especially Pb, S, Sn, Cu and Co are not detected as color determinants.

When we look at the glaze compositions in general, when we look at the examples of navy blue, Light Blue glaze and unglazed tiles, the SiO<sub>2</sub> content, which is the raw material of the tile, is determined at a high rate. This supports the presence of quartz, one of the raw materials of ceramics, in the Well sieved SiO<sub>2</sub> is also glaze making material so it analyzed in high percentage in galzaed samples tile.

When the analyzes made using the in situ analysis technique with the P-EDXRF device in the tomb of Zeynel Bey, which was moved to its new location, and the analysis results of the study conducted by Eskici et al. (2008) are compared, it is seen that the SiO<sub>2</sub> values are compatible in the navy blue and light blue glaze samples. In the Light Blue Glaze samples we examined, unlike the other study, the elements that are color-determining (Pb, S, Sn, Cu) were detected.

The results obtained in this study show that in addition to being a guide in future scientific studies, it can also be a guide in the selection of materials in the restoration and conservation studies that can be done.

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