

A Study on the Colored Glazed Tiles of Soltaniyeh Dome: an Analytical Approach

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ABSTRACT

The destructive tests are limited for investigate the ancient buildings and non-destructive tests or statistical approaches are preferred instead. In this research, the chemical composition of Soltaniyeh Dome tiles was investigated with a statistical approach. Characterization of colored glazed tiles belonging to historical collection of SD was performed using portable XRF, followed by comparison of samples using PCA/Euclidean distance analysis was made. Decorated motifs which were investigated in this study were blue, green, yellow, white and black colors. According to the classification performed by principal component analysis, three groups of glaze composition, consist of bright colors (Group 1), dark colors (Group 2) and sky blue color (Group 3) was recorded. The results indicated the main differences in the chemical composition of samples while having similarities in the existing colors. The reason was related to the different sources of raw materials which has been provided in periods of time. A similarities were observed between the color composition of pottery glazed tiles and the color composition reported on gypsum surfaces by previous researchers. Prog. Color Colorants Coat. 16 (2023), 113-124© Institute for Color Science and Technology.

1. Introduction

Potteries show among the most abundant background in archaeological excavation; archaeologists use potteries to generate statistic conflation, reformulation and classification at the basis of resemblances and diversities among kinds, modes or attributes that are fairly persistence in time and/or geographic areas [1-6].

After the emergence of Islam in Iran architecture, many domes and memorial were constructed in addition of mosques. Between the ancient buildings, it can be mentioned to SD which its utilization and the explanation of its ornamental arrays are the most significant tools for remembering the religious and mental foundations of the architecture of this land and

they are also acceptance for the unfeasible and cultural opinions of leaders of that era [7]. The SD is a remark of the honor and flourishing of the Zanjan city about 700 years ago. In the year 702 AH, emperor "Muhammad Oljayto" decided to construct a building according to the design of the "Qazan khan's" tomb in soltaniyeh. Although the SD is alike to the tomb of emperor "Sanjar" in "Marv", the creativeness and novelty in this building has convert it into one of the wonders of Iranian art and architecture [8]. The SD monument is placed 40 km far from Zanjan city (Figure 1). The upland in which this structure was laid is about 2000 m above the sea level [9].

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Figure 1: Picture of SD located in Zanjan [10].

In the book of Islamic architecture of the Ilkhani period, Wilber was introduced the complete list of colors on the gypsum substrates as follows: white, light blue, dark blue, green, red, orange, yellow, cream, reddish brown and gold. In this regard, it should be noted that most of the research which has been excreted on pigments is visual and in most cases the compounds of pigments have not been identified [11].

There is scanty literature related to the chemical composition of colored glazed tiles of SD monument especially for the interior part of dome. Moreover little research has been done on characterization of interior decor with the aim of maintaining and restoring of it. Most of researches have focused on geometric aspects and visual studies of the decorations, seismic strength and durability of the dome.

Sobouti et al. [12] investigated the decoration of SD. The results of their research have shown that definitely, the dome with its decorative masterpiece, has provided the greatest decorations for the future Islamic Iranian eras. Balilan Asl et al. [13] investigated the numerical concepts and geometric codes hidden in the decoration and architecture of SD. The results show that the concepts and meanings are reflected in the arrays in physical forms (in the form of buildings and the hierarchy of formation of spaces), religious symbols (such as Shamseh, celestial bodies), linear shapes (divine names and verses), colors (to create sense of space). Shirvani [14] studied the pigments which was used in the decoration of exterior porch painting belonged to

SD. The purpose of this study was to identify the color combinations of these decorations. FT-IR and XRF analysis techniques were used for the characterization of elemental and combination of color components. Results showed that, the pigment compounds were red, yellow, black bone and calcium carbonate, which have been used on the plaster bed. Yousefinejad [15] investigated the Turquoise monochrome tiles belonged to SD. In this study, the colorant intermediate elements of glazed samples were investigated by inductively coupled plasma method (ICP) and the constituent phases of the glazed bodies were identified using the X-ray diffraction (XRD) technique. The results show that these glazes in their chemical composition, have more than 3 wt. % of lead element. Also the colorant agent in the Turquoise glazed samples was attributed to the copper element. Sheikh [16] investigated on the restoration of the colored glazed potteries belong to Ilkhany period, excavated at Soltaniyeh Dome. She excreted radiation chemistry for image restoration of these colored potteries. The results showed that the major ingredient of the top layer were Ca and Si in large quantities, which are typical ingredient of glaze. Also results revealed that the glaze of ancient Iranian potteries is alkaline type. This type of glaze is easily scraped and it is chemically instable under wet conditions. As the glaze is affected by moisture, hydrogen ions replace the alkaline ions from the glaze, resulting in the disruption of the structure of the network. Lamahi-Rachti et al. [17], used the proton-induced X-Ray fluorescence (PIXRF) technique for

chemical analysis of archeological glazed tiles belong to various famous Iranian monument such as Blue Mosque, Sardar Mosque and SD. Results showed that the elements such as Si, Al, K, Ca and partly Pb were the major constituent of alkali-lead glazes, whereas other elements such as Co, Cu, Sn and Mn give the characteristic color of the glazes. The chemical composition of the glaze tiles in different part of SD was investigated by Moradi et al. [18]. They used scanning electron microscopy, energy dispersive X-Ray spectroscopy and ICP for characterization of the glazes. Results revealed that cobalt, copper and manganese are the main elements as coloring agents. Holakooei et al. [19] investigated the Persian tilework's belong to safavid eras by wavelength dispersive x-ray fluorescence and then data were managed with principal component analysis (PCA). Simsek et al. [20] have presented a non-destructive method i.e. Portable XRF (PXRF) for analyzing the twenty-five excavated tiles located at the Iznik kiln. The comparison was made by the discussion of characteristic elemental ratios selected from the ceramic technology criteria and PCA/Euclidean distances analysis. They showed that the amount of tin oxide in the glaze decreased over the centuries. Besides, two different types of fluxes were used in the glaze, some containing only potassium, and the others having potassium and calcium. The blue, turquoise, green, red

colors, and black lines were detected in the decors. A copper-iron mixture in the red areas was documented, which reflects the use of bornite.

In this research the chemical composition of colored glazed tiles attributed to the SD, was investigated with a statistical approach for the first time. For this purpose, at first chemical analysis of colored glazed tiles was characterized by PXRF method and then the data was managed by PCA. The objectives of this study is the comparison of colored glazes with different colors and a better understanding the fabrication technology of ancient glazes at the basis of Graph Clustering (GC) method [21, 22].

2. Experimental

2.1. Materials

Twelve colored glazed samples were collected from ancient potteries belonging to the Ilkhanid period from different parts of SD Zanjan, Iran (Figure 2). The mentioned samples are broken pieces or fragments of ancient potteries or walls of the historic building of SD which were provided from culture heritage organization of Zanjan. The appearance of samples were green-blue, light blue, bright blue, light green, bright green, yellow, black, brown and gray.

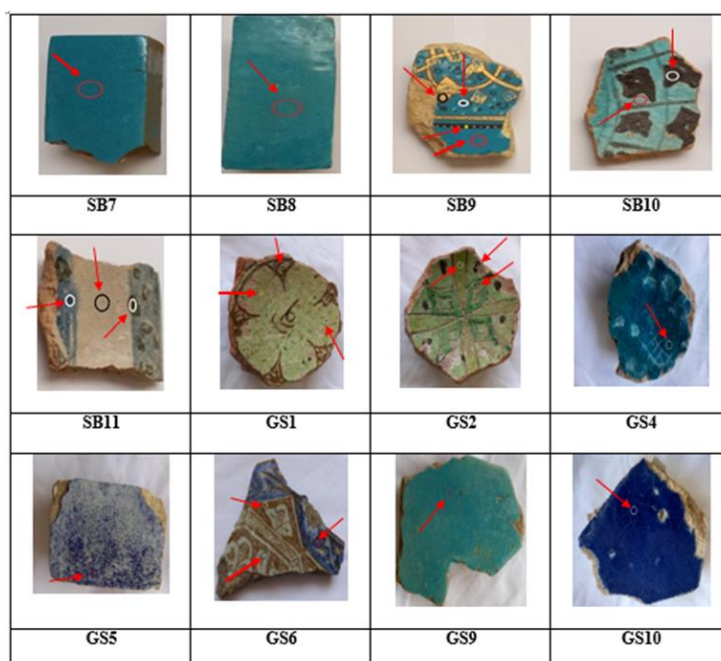


Figure 2: Colored glazed samples from different parts of SD.

2.2. Method

2.2.1. PXRF

Chemical composition of ancient colored glazed samples was investigated by portable X-Ray fluorescence (XRF Niton XL3t), Thermo Scientific company, USA. This type of instrument is conventionally used in characterizing of several archaeomaterial types for instance, pottery [23], bronzes [24], wood [25] and paintings [26].

On the other hand, it is important to note that portable XRF analysis should not be assumed as a replacement to conventional laboratory methods (both chemical and petrographic techniques), but can aid in a primary evaluation a lot of pieces, permitting in simple, rapid and non-invasive way (in principle directly on site) a first grouping of archaeological potteries based on which further chemical and petrographic analysis can be planned [27].

Another decisive feature in evaluating the non-simple results provided by PXRF investigation is related to the data processing; in origination studies, chemical data are usually accomplished by chemometric methods [28-30], as follows PCA [31], dendrograms [32], cluster analysis [33], artificial neural networks [34], etc.

To characterize all the colors of pieces, 24 colored dots (circular area) have been marked on the samples according to Figure 2 and analyzed using PXRF.

2.2.2. PCA

PCA was first suggested by Pearson [35] in 1901. The main objective was to use mathematical alteration to recombine the input original data variables (X) into various “new variables” that can illustrate the main information of the original variables, and these “new variables” were called principal components (PC). These principal components which are separated from each other can efficiently lower the dimension of original variables and improve the modeling speed, so the accuracy of prediction results could be efficiently improved [36]. PCA is a commonly utilized method of statistical analysis when addressing questions of sourcing or provenance [37-41]. Whether the artifact in question is pottery, obsidian, glass or metal, PCA provides a method of analyzing and categorizing compositional data, provided by analyses like XRF, into groups that minimize the intergroup differences while maximizing the differences between groups. It is

a statistical procedure utilized to reduce the number of variables associated with a set of data. In the case of this study, the variables are the various elements detected and measured by the PXRF during the analysis of each glazed color of tiles or potteries.

Principle components are created by optimally weighting the observed variables, in this case the elemental concentrations, in a way that allows for each component created to account for the maximum amount of variation possible. There were actually fourteen components created from the PCA (because there were 14 elements), but most of the components account for small amounts of the overall variation in the dataset and are deemed inconsequential to the identification of color sources such as Cu, Fe, Mn, Ca and Pb. It is important to note that PCA is not a factor analysis because it does not assume that a relationship exists between the observed variables, as in a factor analysis. PCA is merely used to reduce the number of observed variables to see if there are any underlying correlations to be found.

2.2.3. Dendrogram

A dendrogram is a diagram representing a tree. This diagrammatic representation is frequently used in different contexts: In hierarchical clustering, it illustrates the arrangement of the clusters produced by the corresponding analyses [42]. In computational biology, it shows the clustering of genes or samples, sometimes in the margins of heatmaps [43]. In phylogenetic, it displays the evolutionary relationships among various biological taxa. In this case, the dendrogram is also called a phylogenetic tree [44].

2.2.4. Software

A statistical program, titled Minitab 16, was used for drawing graphs of PCA and dendrogram which shows the similarity and differences in the ancient colored glazed samples.

3. Results and Discussion

3.1. Chemical analysis

Fragment colored glazed parts have been examined, but due to the presence of different colors and color changes in some areas of the samples, a total of 24 colored areas have been investigated and analyzed. The results can be seen in Table 1. It can be seen that, various elements

have been identified in specimens. The minimum and maximum variation ranges of detected elements have been shown in Figure 3 a. It is clear that the elements of Pb, Sn, Cu, Mn, and Au have significant values and can be consider as the major elements. The Pb and Sn can be related to the glaze structure but elements like as Fe, Cu and Mn play an important role in creating color in glazes. In between, the Au just has detected for sample SB9-Black revealing that have decorated with gold. In

other samples the concentration of gold is insignificant. The concentration of Cu varies from 0.032 wt. % in the SB11 to a maximum of 5.94 wt. % in the SB8 and for Fe varies from 0.37 wt. % in the SB11 to a maximum of 1.84 wt. % in the SB9 sample. For Zn this range is 0.008 wt. % in GS5 to a maximum of 0.35 wt. % in SB8 sample. Also, the concentration of Mn in the GS2 sample is 0.021 wt. %, which enhances to a maximum of 6.395 wt. % in SB10.

Table 1: Results of PXRF analysis from different areas of colored glazed samples.

sample code	Sb	pb	Zn	Cu	Fe	Mn	Cr	Sn	Co	Ag	Au	As	Ba	Ni
GS1-black	0.04	19.10	0.04	0.26	0.45	0.08	0.11	0.01	0.04	0.01	0.04	0.00	0.06	0.03
GS1-yellow	0.19	38.45	0.10	0.28	0.59	0.04	0.13	0.04	0.00	0.00	0.05	0.25	0.17	0.04
GS2-black	0.01	15.17	0.04	0.30	0.69	0.59	0.10	0.01	0.03	0.01	0.03	0.00	0.06	0.03
GS2-green	0.05	37.77	0.07	0.53	0.49	0.02	0.11	0.06	0.00	0.00	0.05	0.25	0.17	0.03
GS2-yellow	0.04	51.11	0.13	0.46	0.54	0.08	0.15	0.04	0.00	0.00	0.04	0.00	0.22	0.04
GS1-brown	0.01	20.44	0.04	0.15	0.99	0.08	0.11	0.01	0.04	0.01	0.03	0.00	0.05	0.03
GS4	0.01	0.08	0.03	1.14	0.28	0.07	0.08	0.01	0.02	0.01	0.01	0.02	0.01	0.01
GS5	0.01	0.17	0.01	0.04	0.53	0.06	0.04	0.08	0.00	0.00	0.01	0.17	0.37	0.01
GS6-black	0.01	13.78	0.04	0.08	0.46	0.07	0.11	2.62	0.03	0.01	0.02	0.00	0.02	0.03
GS6-white	0.01	12.10	0.03	0.12	0.49	0.07	0.11	2.25	0.03	0.01	0.02	0.00	0.02	0.02
GS6-red	0.01	14.65	0.04	0.12	0.46	0.09	0.12	2.89	0.05	0.01	0.03	0.02	0.03	0.03
GS9	0.01	12.53	0.07	1.74	0.53	0.07	0.10	2.70	0.03	0.01	0.02	0.00	0.03	0.03
GS10	0.02	0.40	0.01	0.03	0.93	0.05	0.04	0.05	0.21	0.00	0.01	0.13	0.43	0.01
SB7	0.04	2.02	0.29	4.47	0.83	0.02	0.21	18.97	0.00	0.00	0.03	0.07	0.55	0.03
SB8	0.04	1.35	0.35	5.94	1.19	0.03	0.16	21.13	0.00	0.00	0.04	0.06	0.65	0.03
SB9-black	0.01	10.57	0.04	1.38	0.92	0.08	0.11	3.25	0.04	0.03	4.90	0.00	0.03	0.03
SB9-Yellow	0.03	18.98	0.07	2.16	1.15	0.04	0.13	8.15	0.00	0.00	0.03	0.16	0.09	0.03
SB9-Blue	0.03	17.36	0.06	2.13	0.39	0.05	0.13	7.58	0.00	0.00	0.03	0.19	0.09	0.03
SB9-White	0.08	15.64	0.04	2.14	1.84	2.01	0.17	4.16	0.05	0.01	0.05	0.02	0.04	0.08
SB10-White	0.01	0.03	0.03	1.00	0.63	6.40	0.07	0.01	0.03	0.01	0.01	0.03	0.03	0.02
SB10-Red	0.01	0.03	0.03	1.31	0.71	0.05	0.08	0.00	0.09	0.01	0.01	0.00	0.02	0.02
SB11-Blue	0.02	0.01	0.03	0.06	1.12	0.04	0.03	0.02	0.10	0.00	0.01	0.01	0.01	0.01
SB11-Black	0.02	0.02	0.02	0.06	0.78	0.04	0.03	0.01	0.00	0.00	0.01	0.02	0.50	0.01
SB11-Green	0.02	0.02	0.01	0.04	0.37	0.10	0.05	0.00	0.05	0.04	0.01	0.03	0.49	0.01
Min.	0.01	0.01	0.01	0.03	0.28	0.02	0.03	0.00	0.00	0.00	0.01	0.00	0.01	0.01
Max.	0.19	51.11	0.35	5.94	1.84	6.40	0.21	21.13	0.21	0.04	4.90	0.25	0.65	0.08

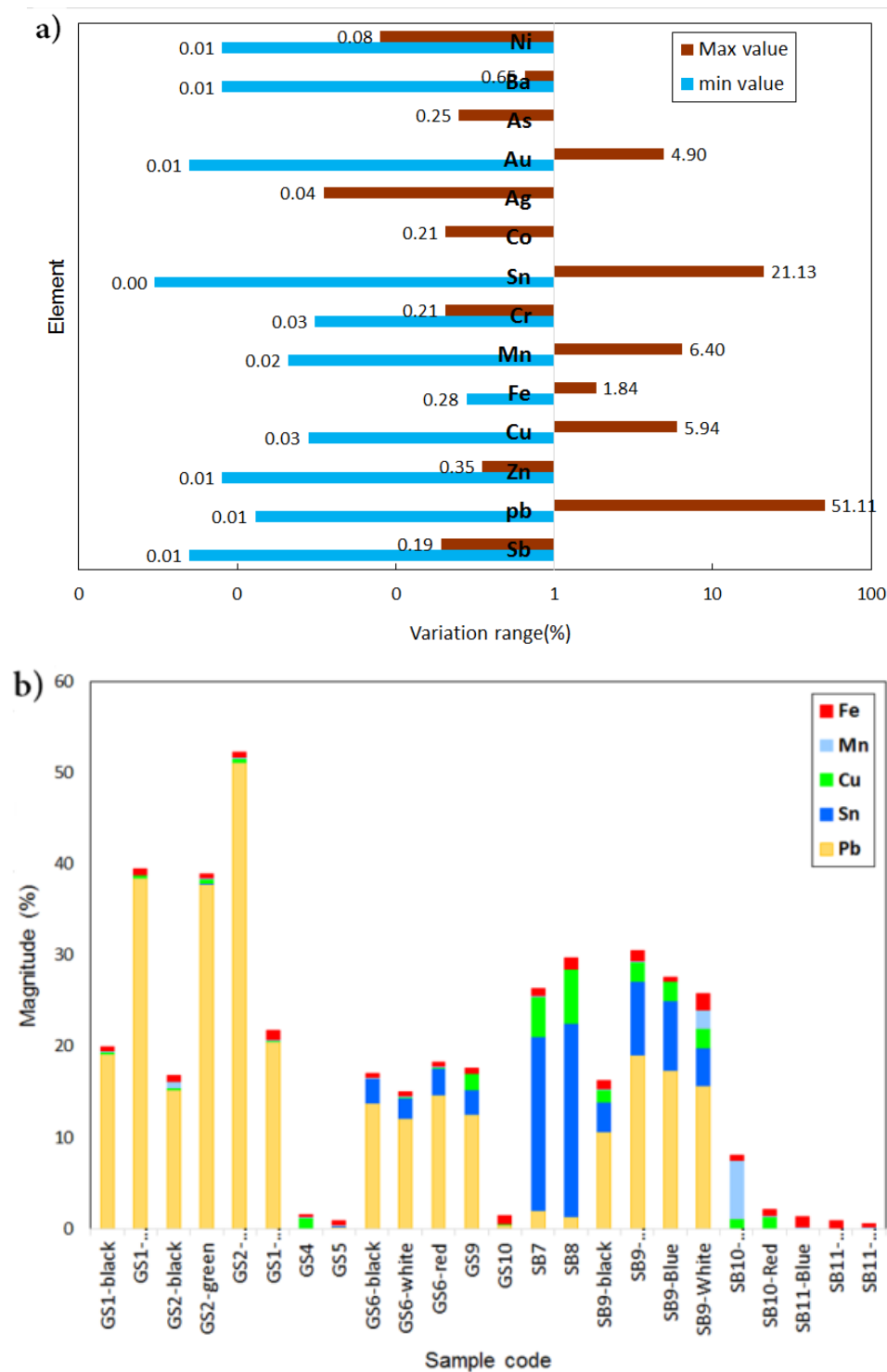


Figure 3: The min. and max. variation ranges of detected elements (a), The variation ranges of the major elements (b).

The variation range for the main detected elements can be seen in Figure 3 b. It is clear that Fe can be seen in all samples and Cu in most of them, revealing that they play effective role as colorant agent in the glaze.

Another important point regarding the concentration of elements is related to Pb and Sn. For Pb in the sample of GS4, the minimum concentration of 0.084 wt. % increases to high value of 38.44 wt. % in GS1.

Generally, its usage was customary for decreasing of melting point (flux agent) of glaze and for brilliant appearance of glaze surface. On the other hand, the Sn from 0.003 wt. % in the SB11 changes to 21.13 wt. % in the SB8 sample, revealing a high variation range. Utilization of Sn (as SnO_2) in glaze as opacifier is common and its history back to centuries ago. The magnitude of it is related to the glaze composition and firing temperature. The ratio of Sn/Pb has been shown in Figure 4. It can be seen that the magnitude of this ratio varies from 0-1.3 with average of 0.24 which has conformity with results of Simsek et al [20]. This ratio for Sb-7 and SB-8 is 9.4 and 19.6 respectively which is strangely high and was not take into account in Figure 4. It seems that the technology for formulation of glaze for Sb-7 and SB-8 samples is different from the others.

According to the results of portable PXRF analysis on the samples of the present study, it is observed that these ancient colored glazes can be considered as lead alkaline glazes that have been opacified by tin oxide which is consistent with the results of previous research in this area [12].

3.2. Statistical analysis

The score plot graphs the scores of the second principal component versus the scores of the first principal component. If the first two components account for most of the variance in the data, one can use the score plot to assess the data structure and detect clusters, outliers, and trends. Groupings of data on the plot may indicate two or more separate distributions in the data. If the data follow a normal distribution and no outliers are present, the points are randomly distributed around zero. The Score Plot involves the projection of the data onto the PCs in two dimensions. The PCs were computed to provide a new space of uncorrelated 'variables' which best carry the variation in the original data and in which to more succinctly represent the original 'samples'.

In this study, PCA has been applied to those samples that have different and higher concentrations of chemical elements than other samples, the results of this analysis are shown in Figure 5 a. According to this Figure, three categories is obvious. In this way, SB7 and SB8 samples are completely different from the others. These samples are very similar in terms of color (sky blue) and on the other hand, the concentration of elements are close in terms of chemical composition.

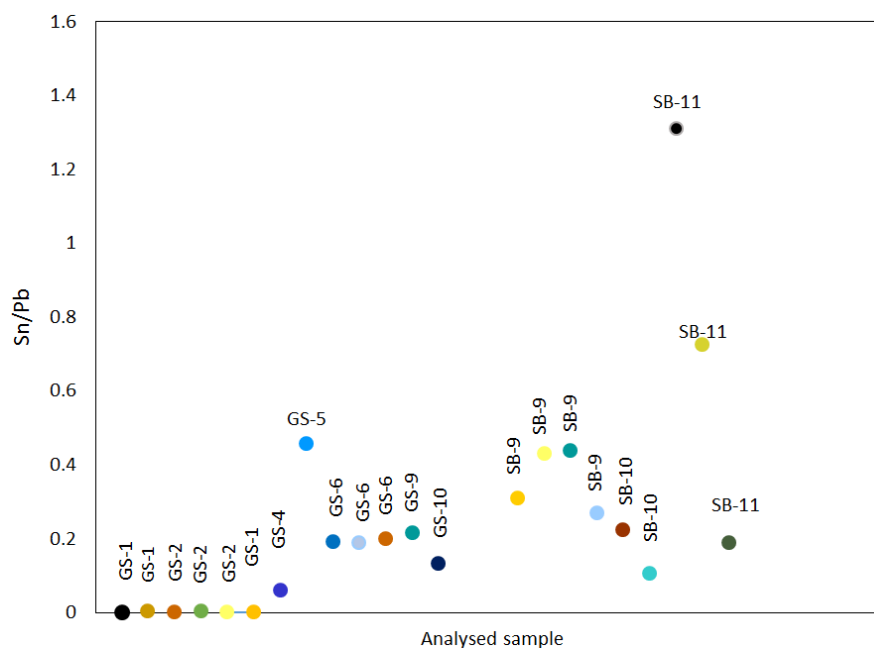


Figure 4: Evaluation of Sn/Pb in the analyzed samples.

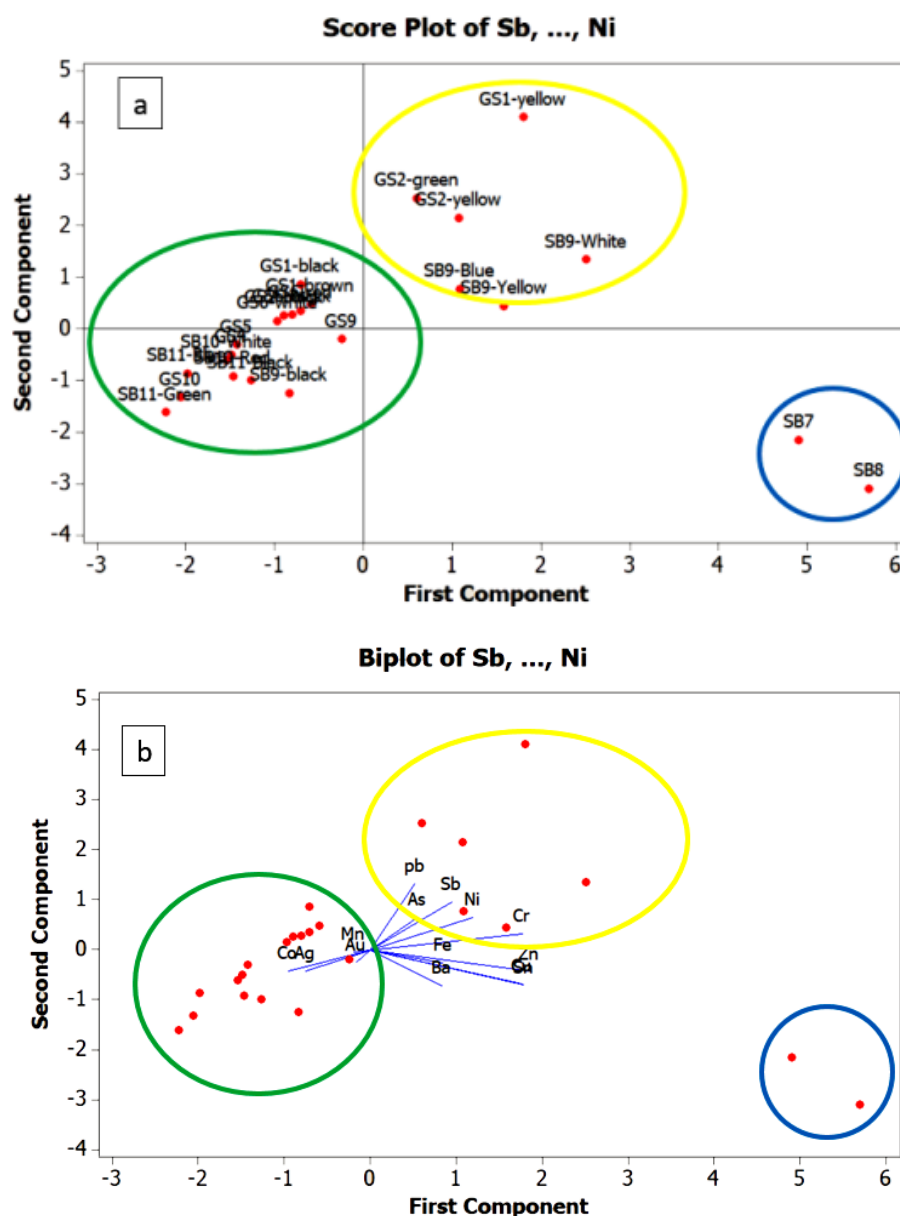


Figure 5: Score Plot of the first component versus second component (a). Biplot of Elements: Sb, Ni (b).

The magnitude of Sn in samples of SB7 and SB8 is 18.97 wt. % and 21.13 wt. % respectively. For Pb element, the magnitude in each of them are about 2.01 and 1.35 wt. %. In this regard, the weight percentage of Zn is about 0.28 % and 0.35. The concentrations of Cu and Fe in these two samples are about 4.47 and 5.94 % and 0.83 and 1.18, respectively. Other elements such as Mn, Cr, Co, etc., have concentrations close to each other.

Beside, samples of GS1-Yellow, GS2-Green, GS2-Yellow, SB9-Blue, SB9-White and SB9-Yellow are also considered as the next category. Samples with yellow colors look the same and samples with black or

brown or darker colors are in another category. On the other hand, it is observed that the samples with light colors are completely separated from the samples with dark ones.

Feizi, et al. [45] have reported the colored glazed tiles discovered in the Castle Hill from the Soltaniyeh Plain in Zanzan with a variety of light blue, deep blue, turquoise, green, gray, and black colors. These specimens are similar to the samples of colored glazed pottery of the current study. The variety of color for colored glazed pottery related to the SD has been reported in another study too [11] which is matching with our results. On the other hand, it has been

observed that although in some samples, the colors are similar, but the difference in concentration in some elements is so obvious. These results can be considered as a pattern for glaze samples in Zanjan region.

Biplots are a type of exploratory graph used in statistics, a generalization of the simple two-variable scatterplot. A biplot allows information on both samples and variables of a data matrix to be displayed graphically. Samples are displayed as points while variables are displayed either as vectors, linear axes or nonlinear trajectories. In the case of categorical variables, category level points may be used to represent the levels of a categorical variable. A generalized biplot displays information on both continuous and categorical variables.

Figure 5 b shows Biplot of Elements of Sb... Ni. It indicates the weight and effect of each variable in the PC. In this case, the variables are the elements. According to the results of Figure 6, it is obvious that two elements of Co and Ag, have a negative correlation with PC1 and PC2 that is about 1.75 respectively and 2.2 for PC1 and about 1.7 and 1.8 respectively for PC2. On the other hand, Zn, Cr, Cu and Sn have the most correlation with PC1 revealing that these elements are so important and effective in PC1. It can be seen that two elements of As and Pb have the least correlation

with PC1. Also, it is observed that elements of Pb, Sb and As have the most correlation with PC2 and it means that these elements are so effective. On the other hand, Figure 5 b, Displays the coefficient of each of the variables in PC. It can be seen that, in the left category, most of specimens are located and has darker colors. Elements such as cobalt, silver, gold and manganese are more important and effective in this category. While in the middle category, which has a less number of ancient specimens has lighter colors. It can be seen that the elements such as Pb, Ni, As and Sb have more effects on glazed specimens. It is also observed that SB7 and SB8 samples, which are in the right category, have the highest correlation with two important elements of Cu and Zn.

A dendrogram is a visual representation. Specifically, it is a tree or branch diagram where there are many elements at one end, and few, or one, at the other. The branches represent categories or classes and the diagram implies an order or relationship between these categories or classes. The members of these categories are similar in some fashion or have a number characteristic in common. Another name for these categories or classes is cluster. And the process of placing the items into a specific cluster is known as clustering [46].

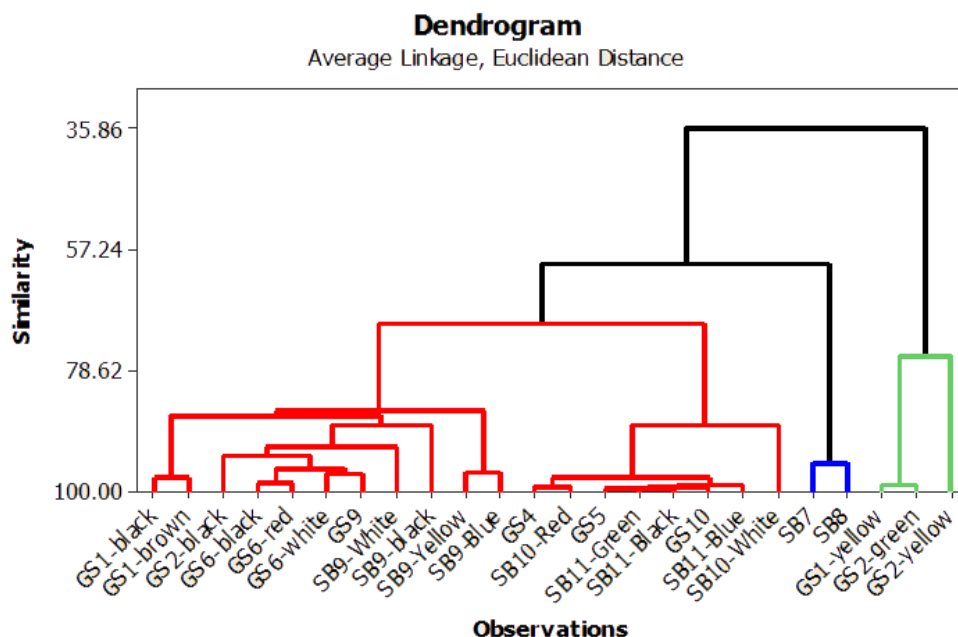


Figure 6: Dendrogram of the similarity on the composition of ancient colored glazed samples.

In Figure 6, dendrogram of the similarity on the composition of colored glazed samples is presented. As it can be seen in the present dendrogram diagram, the clustering of the present ancient specimens composed of three categories and on the other hand, there are a number of subcategories that have shown an important result. This graph obviously demonstrates that two colored glazed samples of SB7 and SB8 and on the other hand two colored glazed samples GS1 and GS2 are very different from the other samples. As can be seen, the ancient samples that are closer to each other, have similar properties and chemical composition to each other, and those samples that are more distant to each other, will have different properties and chemical composition. According to the Fig.6, it is observed that the three samples GS2-Black, SB9-Black and SB11-Black, while having black colors, but their similarity in terms of chemical composition are low and are more different from each other. On the other hand, two samples SB9-Black and SB11-Black, while having a black color, are different from the GS6-Black sample in terms of chemical composition and properties.

Researchers have shown that the decorations of the Ilkhanid period (SD) have an independent and unique style [2]. On the other hand, their results show that the decorations related to the Ilkhanid period, spread in a wide geographical area and then transferred to other areas. According to these results, the difference in concentration and chemical composition of colored glazed tile samples related to SD, while similar in color to these samples, can be reasonable.

4. Conclusion

An analytical study with statistical approach was performed on the ancient colored glazed potteries related to SD. The results of this research show that the grouping of these ancient samples can be categorized in three groups according to the glaze composition. From aspect of color, it was shown that the color can be divided to three groups. i.e. samples with bright colors (Group1), with dark colors (Group2) and with sky blue colors (Group3). Samples of Group1 are rich in Pb, Mn and Cu, Group2 are rich in Fe and Mn and the Group3 rich in Cu and Zn. Different behavior of samples SB7 and SB8 (Group3) compared to other samples, which may be due to the different origins of the raw materials used in their manufacturing process and another reason, could be due to the fact that they belong to later time periods than other samples. It was shown that the Mn, Cu and Fe play effective role for color of the Soltaniyeh Dome tiles. The reason for the observed differences in the chemical composition of the elements in the colored glaze samples of the present study, while having similarities in the existing colors. A similarities were observed between the color composition of pottery glazed tiles and the color composition reported on gypsum surfaces by previous researchers.

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