

Chemical Composition and Manufacturing Technology of a Collection of Various Types of Islamic Glazes Excavated from Jordan

Ziad al-Saad*

Institute of Archaeology and Anthropology, Yarmouk University, Irbid-Jordan

A collection of Islamic glazed pottery shards that were excavated from the archaeological site of Dohaleh/Northern Jordan were chemically analysed. The glazes belong to three different decorative styles. The chemical analysis of the glazes was carried out using energy dispersive x-ray fluorescence. The chemical analysis results enable the classification of the glazes into the three distinct compositional groups with reference to their principal modifier, these are: the alkaline glazes, the high lead glazes and the lead–alkali glazes. In some cases the body fabric was analysed by a combination of petrographic and chemical analysis techniques. The study show that inherited traditional techniques were combined with innovative Islamic techniques were used for the production of the glazes.

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Introduction

he date of the first man-made glaze is not known for certain. However, there is strong evidence that it originated from the Near East some time around 4000 BC (McCarthy, Vandiver & Gibson, 1995). In the first instance glaze was applied to stones such as steatite and quartz. Glaze on faience soon followed utilizing a similar technology to that of glazed stones. Approximately 2500 years is believed to have passed after the introduction on faience before glaze was used on clay substrate material.

The first examples of glazes on pottery vessels are dated to the 15th century BC, about the same time that glass vessels are believed to have developed (Hamer, 1975: 145; Lilyquist & Brill, 1993; Moorey, 1994; McCarthy, Vandiver & Gibson, 1995).

From 1500 BC onwards the glaze industry developed in many respects and various formulations of alkaline and lead glazes were used in various parts of the world. The Romans spread the secrets of glaze making throughout their Empire. From Rome the idea of lead glaze on pottery spread to Han China where the combination of it with the porcelain materials already known resulted in wares of a remarkable richness and quality. The existing recipes served the potters of the Byzantine Empire throughout its duration and from there spread back into Europe where they survived without much alteration up to the 18th century (Frierman, 1970).

The Muslims inherited glaze making traditions from their predecessors. In addition, a number of innovations occurred. Lead-alkali-silicate and lead-silicate glazed wares were introduced along side the alkalisilicate glazes in the early Islamic period. At the time of the rise of Islam in the 7th and 8th centuries a small amount of lead glaze ceramics was made in the Near East, in Egypt and Byzantium. The prevalent ceramic tradition was one in which relief-decorated unglazed slipware predominated. The Muslims revived the old glaze tradition and gave it strong impetus with perfection and innovation. The glaze industry flourished during the Abbasid period when brilliant developments of ceramic art and technology were achieved. Different glaze formulations of lead glazes and alkali silicate glazes were used to produce local imitations of the wonderful Chinese T'ang ceramics (Frierman, 1970; Kleinmann, 1986).

The Seljuk Turks introduced a new school of pottery making. They made what may be the most colourful and beautiful ceramics made outside of the Far East. Lustre was raised to a high art in Iran, especially in Kushan. The old T'ang three-colour wares were reinterpreted and faience reappeared in new forms. However, this dramatic flourishing was to be short lived. During the first half of the 13th century the Mongols swept in from Central Asia and this caused the discontinuation of the most sophisticated types and techniques. A slow but persistent artistic recovery occurred in the late 13th and 14th centuries; the most symptomatic ware was the blue or black painted wares, which were covered with a transparent glaze that ran the colours a little and had a crackle, echoing the work of the Chinese potters. Although lustre and other overglazed ceramics were made again, both the pots

^{*}Associate Professor of Archaeometry and Conservation.

and techniques became widely diffused (Frierman, 1970; Perez-Arantegui, 1995).

In this work, various types of Islamic glazes that were excavated from Jordan were chemically analysed in order to reconstruct their manufacturing technology. The contribution of Islamic glaze makers to the inherited glazing techniques was specially emphasized.

Materials and Methods

Archaeological context of the samples

In the period between 1990-1993 major excavation works were conducted by a joint team from the Institute of Archaeology and Anthropology/Yarmouk University and the Department of Antiquities of Jordan at the archaeological site of Dohaleh/northern Jordan. The site is situated in a fertile area 25 km to the southeast of the city of Irbid (Figure 1). The excavation works unearthed large quantities of artefacts and architectural remains. The foundations and structural remains of a mosque, a Byzantine church and large numbers of houses were uncovered. Also a large Islamic cemetery was discovered. Large quantities of various styles of pottery, glass, coins and metal objects were also unearthed from the site, especially from the cemetery. The archaeological evidence extracted from the remnant structures in addition to the stylistic and typological studies of pottery and coins strongly suggest that the site was continuously occupied from the early Roman period to the Ottoman (Sari, 1991,

A great deal of ceramic materials, including a number of vessels, in addition to large amounts of pottery shards were uncovered from the residential area of the site. Various styles of Roman, Byzantine and Islamic pottery were identified. The most abundant among these styles is the Islamic painted pottery. The largest proportion of this pottery was dated to the Ayyubid/Mamluk period based on the morphology and decorative styles. In addition to the painted pottery a relatively small amount of glazed pottery shards were uncovered from the same level. The glazed shards were attributed to the Ayyubid/Mamluk period based on their archaeological context and the associated materials, mainly coins (Sari, 1992b).

Samples

Unfortunately, when Islamic glazed ceramics are studied, only a few objects are available for analysis because glazed fragments are scarce at archaeological sites. Taking into account this limitation, 17 glazed shards of different glaze types were selected for this study. The selected shards were derived in most cases from recognizable objects such as jugs, jars, and bowls. The shards were selected to represent the different styles of glazes found at the site. Three major styles were identified, these are:

1—Coloured monochrome-glazed shards. This type of glaze is characterized by having a transparent glaze layer on a slipped coarse earthenware. The glazes have turquoise and blue as their most common colours. The shards show incised decoration under the glaze layer. Seven shards of this type were selected for the chemical analysis.

2—Underglaze painted shards. In these glazes the design was executed directly on the body fabric using a mixture of unfused coloured minerals. The decoration was painted either in black or blue. The paint layer was sealed *in situ* by a coating of translucent glaze mainly of green colour. Six shards from this type were selected for the chemical analysis.

3—Blue in-glaze decorated shards. These glazes are characterized by their opaque white background with an overlaying blue decoration. The decorations consist mainly of geometric motifs. Four shards from this type were selected for the chemical analysis.

Analytical techniques

The chemical compositions of the glaze samples were determined by energy dispersive x-ray fluorescence analysis. Calibration was done using a variety of synthetic and natural mineral standards. The glaze compositions were determined by averaging results of at least five spot analyses at random points of the glaze surface.

Body samples were examined by standard petrographic methods, using polarizing light microscope and a point counter. Chemical analysis of the body samples was done using atomic absorption spectrometry.

Results and Discussion

Classification of the glazes

Glazes may be described and classified in many ways; for example, by their maturing temperature, their principal modifier or flux and the wares on which they may be used (Rice, 1987: 90; Hodges, 1976). The most informative of these classification systems is the one that is based on principal modifier which is the one that will be used in this study.

The glazes under study can be classified with reference to their principal modifiers into three distinct groups; these are: (1) the alkaline glazes (Table 1), (2) the high lead glazes (Table 2), (3) the lead–alkali glazes (Table 4). For each type the chemical composition, raw materials used manufacturing technology will be discussed.

Chemical composition and manufacturing technologies I—Alkaline glazes. All the glazes in this group are of the coloured monochrome. The glazes are characterized by having significant amounts (average %

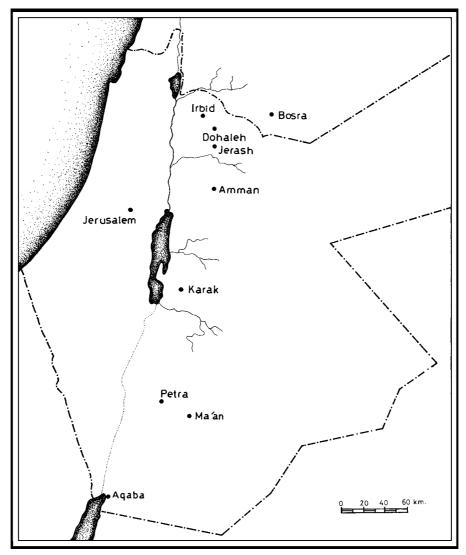


Figure 1. Site map.

 $Na_2O = 16.02$) of soda which was used as the main flux. The glazes also contain appreciable amounts of lime, potash and magnesia as depicted in Figure 2.

The general molecular formula of the glazes calculated (Green, D., 1978) based on the average percentage values of the oxides (Table 1) is the following:

 $Na_2O = 0.58$

 $K_2O = 0.05$ $\overline{\text{CaO}} = 0.25$

MgO = 0.12

 $Al_2O_3 = 0.04$

 $SiO_2 = 2.45$

Obtaining precise recipes based on the calculated formula is fairly difficult. This is due to the fact that analysis of fired glazes does not take into account the loss of alkali through volatilization during firing, the high take up of silica from the body and the presence of impurities in common sands or alkalis derived from the

distillation of plant ashes. However, some general inferences about the nature of the raw materials can still be obtained.

The formula indicates that silica may have come from using siliceous sand or flint. Soda may come fro either plant ashes or natron. The presence of appreciable amounts of lime, potash and magnesia suggests that plant ashes were used.

As the plant ashes are soluble in water, they must be pre-fitted with silica along with other minor ingredients to prepare the glaze suspension. This suspension may have been added to the clay body by various ways mainly brushing or dipping. The uniformity of the glazes in terms of their colour and composition suggests that fritting process was used in their manufacture.

Alkali glazes would have posed a serious technical challenge to the glaze maker due to their high solubility and poor adhesion to the clay body. The high alkaline

Table 1. Chemical composition of the alkaline glazes

Object no. Type Colour	1 Bowl sherd Purple	2 Frag. of a jar Turquoise	3 Frag. of a jug Turquoise	4 Rim sherd Sky-blue	5 Handle frag. Blue	6 Handle of a jar Blue	7 Rim sherd of a jar Purple	
% Oxide								Average
SiO ₂	69.01	66.98	65.39	66.35	66.55	65.94	67.05	66.75
Na_2O_3	14.62	14.34	17.26	15.92	16.01	17.03	16.93	16.02
K_2	2.10	2.62	2.01	2.20	2.09	2.00	2.03	2.15
CaO	6.26	6.72	6.14	6.43	6.62	6.32	6.83	6.47
MgO	2.11	2.34	2.03	2.16	2.09	1.98	1.99	2.10
PbO	_	_	_	_	_	_	_	_
Al_2O_3	2.16	2.10	1.92	1.74	1.92	1.89	1.99	1.96
MnO	2.14	_	_	_	_	_	1.92	_
Fe_2O_3	1.16	1.12	1.33	0.92	1.42	1.46	1.11	1.22
CuO	_	3.42	3.62	4.02	2.96	2.72	_	_
CoO	_	_	_	_	_	_	_	_
_	0.04	_	_	0.03	_	_	0.03	0.03
ZnO	_	0.06	0.064	0.073	0.052	0.056	_	_
Total	99.60	99.70	99.76	99.84	99.71	99.43	99.88	

Table 2. Chemical composition of the high lead gazes

Object no. Type of object Colour	8 Bowl sherd Transparent, dark green	9 Rim sherd Transparent, green	10 Fragment of a jug Transparent, brown	Fragment of a jar Yellowish green	12 Handle of a large jar Dark brown	13 Handle fragment Pale green	
% Oxide							Average
SiO_2	31.00	32.08	30.10	29.21	28.52	30.36	30.21
PbÕ	57.30	56.30	56.76	59.10	59.42	58.26	57.86
Na ₂ O	0.51	0.59	0.46	0.56	0.45	0.41	0.50
$\tilde{K_2O}$	0.32	0.30	0.48	0.21	0.31	0.38	0.33
CaO	1.51	1.56	1.50	1.70	1.28	1.62	1.53
MgO	0.30	0.36	0.40	0.41	0.52	0.31	0.38
Al_2O_3	6.70	6.60	6.36	6.70	6.96	7.10	6.74
MnO	_	_	2.12		1.92		
Fe_2O_3	0.40	0.46	1.20	1.89	0.20	0.65	0.80
CuO	1.60	1.49	0.20		0.06	0.52	
SnO_2	_	_	_		_	_	_
$Ag ilde{O}$	0.31	0.21	0.12	0.13	0.24	0.30	0.22
Total%	99-95	99.95	99.50	99-91	99.88	99-41	

Table 3. Chemical composition of the glaze layer and the body (Glaze composition was recast to 100% after subtraction of the lead content)

	Average %SiO ₂	Average %Na ₂ O	Average %K ₂ O	Average %CaO	Average %MgO	Average %Al ₂ O ₃	Average %Fe ₂ O ₃
Glaze	74·20	1·23	0·81	3·76	0·93	16·56	1·96
Body	67·38	3·62	1·65	8·39	2·16	12·65	3·10

content of the glaze has the advantage of lowering the maturing temperature of the glazes making them suitable for application to earthenware. On the other hand, the high alkali content means that the glazes have expansion coefficients which are significantly greater than those of earthenware bodies. This results in making the glaze highly susceptible to "crazing". Crazing occurs when the glaze contracts more than the body and develops a network of surface cracks. No signs of crazing are observed on the glazes under study.

Table 4	Chemical	composition	οf	the	load	alkali	alazes
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Number Type Colour	14 Body shard		15 Rim shard		16 Handle shard		17 Body shard		Average	
	White	Blue	White	Blue	White	Blue	White	Blue	White	Blue
% Oxide										
SiO ₂	44.13	52.44	42.90	49.24	45.02	49.46	46.01	52.45	44.51	50.90
Na ₂ O	7.20	5.26	7.31	5.26	7.62	5.43	8.42	5.89	7.64	5.46
K_2 O	1.10	1.89	1.05	2.10	1.62	1.76	1.11	1.92	1.22	1.92
CaO	8.13	4.26	7.63	5.26	7.81	5.78	7.02	4.89	7.65	5.05
MgO	0.6	1.21	0.51	1.10	0.58	1.26	0.59	1.02	0.67	1.15
PbO	31.34	24.65	32.20	26.45	30.30	25.54	29.09	23.65	30.73	25.07
Al_2O_3	1.12	2.10	1.33	2.31	1.21	2.70	1.32	2.61	1.25	2.43
Fe_2O_3	1.30	3.20	1.22	3.42	0.92	3.10	0.87	2.82	1.08	3.14
CuO	0.34	1.78	0.67	1.86	0.34	1.76	0.43	1.87	0.45	1.82
SnO_2	4.51	1.23	4.87	1.12	4.26	1.32	4.78	1.10	4.60	1.19
AgO	0.21	0.12	0.23	0.14	0.30	0.11	0.26	0.16	0.25	0.13
CoO		0.91		0.86	_	0.90		0.84		0.88
NiO	_	0.32	_	0.38	_	0.39	_	0.30	_	0.35
ZnO	_	0.43	_	0.34	_	0.38	_	0.40	_	0.39
Total	99.98	99.80	99.92	99.84	99.96	99.89	99.90	99.92		

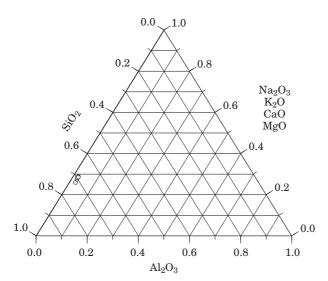


Figure 2. Ternary diagram showing chemical compositions of alkaline glazes (Table 1). The concentrations are normalized to 100%.

This is because the glaze makers overcame the fitting problem of the alkaline glazes by separating the glaze layer from the clay body by a highly siliceous slip. All the monochrome glazes selected for the study had been slipped which indicates that this was a technique adopted when working with alkaline glazes. Manganese and copper were the main colourants used in the glazes. The manganese oxide produced a rich purple colour while the copper produced the characteristic sky-blue azure.

2—High lead glazes. This type of glaze was used to produce the underglaze variety. As shown in Table 2 and Figure 3 the glazes have lead as a principal

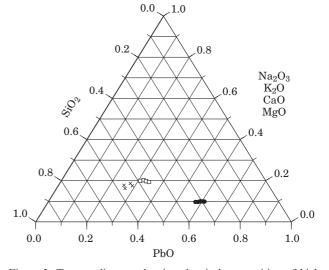


Figure 3. Ternary diagram showing chemical composition of high lead glazes (Table 2) and lead-alkaline glazes (Table 4). The concentrations are normalized to 100%. ●, High lead glazes; □, lead–alkali glazes—white layer; X, lead-alkali glazes—blue layer.

modifier with an average content of 57.86%. They have an alkali average content (Na₂O+K₂O) of less than 1% and an aluminium average content of 6.74%. The glazes have the general molecular formula of:

PbO=
$$0.85$$

(KNa)₂O= 0.04
CaO= 0.08
MgO= 0.03
Al₂O₃= 0.22
SiO₂= 1.6

Glazes of this formula may have been produced by more than one route. In the simplest method, lead glazes may be applied by simply brushing the surface

of the vessel with a powdered lead compound such as the black litharge (PbO), or galena (PbS), or red lead (Pb₃O₄). Alternatively, a suspension of a lead compound with silica in the form of quartz sand, ground quartz or chert pebbles, with little slip is applied to the clay body either by brushing or dipping. A third possible method is by preparing a frit by firing together the lead compound and silica together.

It has been proved that the interaction between the glaze mixture and the body may give some clues on the method used for producing the glaze (Hurst & Freestone, 1996; Tite et al., 1998). When a powdered lead compound is applied to the surface of the vessel, the lead diffuses into the body during firing, talking the alumina and silica required to complete the glaze composition from the body. This means that if the lead oxide content is subtracted from the glaze composition and recast the composition to 100%, the resulting composition should match that of the body. When lead-silica mixture is used instead, the lead and silica will react together during the firing process to form the glaze. Some of the body components like the alkalis, calcium and aluminium will diffuse to and react with the glaze. In this case it is more likely that the composition of the glaze will differ from that of the

The chemical composition of the body used in these glazes does not match that of the glaze as shown in Table 3.

This is a strong indication that lead–silica mixture probably with some other minor ingredients was used in the production of the glazes rather than a powdered lead compound alone.

The glazes contain an appreciable amount of alumina with an average content of 6.74%. The question is the source of this alumina whether it was deliberately added to the glaze suspension or came from the body through diffusion. It has been found that when a lead-silica mixture is used, the alumina content of the glaze is likely to be due, at least in part, to diffusion from the body (Rye & Evans, 1976; Morlea et al., 1997; Tite et al., 1998). However, the significant amounts of alumina found in the glazes suggest that alumina may have been deliberately added to the glaze suspension in a form of clay. The glaze makers may have been aware of the advantages of adding alumina to their glazes. Alumina has the advantages of controlling the viscosity of the glaze suspension and prevents it from being too runny, strengthening the glaze in firing.

In order to ensure a successful firing at relatively low temperatures, the glazes were formulated to have a composition close to that of the eutectic (i.e. % SiO₂=31·7, % PbO=61·2, % Al₂O₃=7·1) (Tite *et al.*, 1998). Based on their composition, the maturing temperature of the glazes is in the range of 800–1050°C (Cooper & Lewenstien, 1998: 41–59). This temperature is suitable for glazing earthenware bodies and was technically attainable by the kilns used by the Islamic potters.

Application of high lead glazes to clay bodies is easier with less problems in comparison to alkaline glazes. The molten lead glazes have relatively low surface tension (Bansal & Doremus, 1986) which means better "wetting" properties and thus the risk of glaze "crawling" is significantly less. In addition, the high lead glazes have a low thermal expansion coefficient that match those of earthenware bodies. This means that they have good fitting ability to the clay body and they fire with few flaws (Tite et al., 1998). Lead glazes have also the advantage of relative cheapness due to the ease of their preparation and firing and due to the abundance of lead ores in the Islamic World. Furthermore, high lead glazes are characterized by their brilliance due to the higher spectacular reflectance from the glaze surface which results from their high refractive indices. These altogether made lead glazes quite popular during the Islamic period.

The glazes have a relatively high and variable silver content (% Ag=0·13–0·3). Silver should have been incorporated into the glaze with lead ores used in the production of the glazes. The significant amount of silver indicates that some of the used lead ores were not de-silvered.

Manganese and copper were mainly used to colour the glazes. The manganese produced the brown colour while copper produced the green and yellowish green.

Lead-alkali glazes. Lead-alkali glaze was used to produce the charming blue on white glazes. The decoration of white vessels with cobalt-blue designs, sometimes called blue in-glaze decoration has a long pedigree going back to the 2nd millennium BC (Kleinmann, 1986). The major innovation of the Islamic potters was the use of tin-opacified white glaze as background (Mason & Tite, 1997). The first wares thus decorated appeared only in the Abbasid period (Perez-Arantegui, 1995). The chemical analysis results show that both the blue and the white glazes were made of lead-alkali glaze (Figure 3). However, there are some compositional differences between the two in terms of their lead contents, the colourants and opacifier used in each glaze.

Opaque white glaze

The general molecular formula of these glazes calculated based on the average percentage of oxides (Table 4) is:

 $\begin{array}{c} PbO = 0.32 \\ Na_2O = 0.29 \\ K_2O = 0.03 \\ CaO = 0.32 \\ MgO = 0.04 \\ Al_2O_3 = 0.03 \\ SiO_2 = 1.73 \\ SnO_2 = 0.07 \end{array}$

The recipe for glaze of such formula should have involved mixing a lead compound and silica with the addition of tin oxide and alkali most likely in the form of a plant ash. The addition of alkali to lead glazes improves the hardness of their surfaces and clarifies their colour. However, because alkali is soluble in water it must have been pre-fritted with silica before preparing the glaze suspension.

The significant amounts of tin unequivocally indicates that tin was deliberately added for its opacifying effect. This is mentioned in abu'l-Qasim's 14th century treatise on ceramics (Allan, 1973). Among matters mentioned in the treatise he describes the preparation of a pigment for white glaze "lead and tin were melted together until they form a white substance to which quartz and plant ashes were added to make a glaze frit". The melting of lead and tin together suggested by abu'l-Qasim has a technical reason as demonstrated by modern research results (Kingery & Vandiver, 1986: 116). Heating tin alone will result in incomplete oxidation of tin due to the formation of a coating of tin oxide. However, with the addition of lead, the formation of tin oxide coating is prevented by intermixture of the tin oxide with the lead oxide.

The glazes have a significant lime content (average % CaO=7.65). Although lime has the advantage of reducing lead solubility it is more likely that it came as unintentional addition with the plant ashes used as a source of alkali rather than a deliberate addition. The conscious use of lime can hardly have occurred during the Islamic period. It was always present with the plant ashes and its properties will have been taken into account unconsciously, along with the other ingredients of these raw materials, in selecting for particular glazes.

The interaction between the body and the glaze can provide some useful information about the method used in the glaze application and firing (Morlea, 1996; Tite et al., 1998). The body used for the lead alkali glazes was made of calcareous clay with a CaO average percentage of around 20%. It is expected that if the glaze suspension was applied to unfired body, this would result in releasing carbon dioxide due to the decomposition of the calcium carbonate upon firing. This would have resulted in the formation of trapped glaze bubbles in the glaze layer. The absence of such bubbles from the glazes suggests that the glaze suspension was applied to biscuit-fired bodies rather than to unfired bodies.

There are other advantages of using calcareous body in producing lead alkali glazes. Calcareous bodies have the advantage of having thermal expansion coefficients that closely match that of the lead-alkali glazes. Furthermore, calcareous body fires to a buff colour in an oxidizing atmosphere which means that with that body white glaze can be produced at lower concentrations of tin oxide than would be necessary with a non-calcareous clay body (Tite et al., 1998). The selection of this particular type of body with this

particular type of glaze indicates that the Islamic potters knew these advantages and consciously chose the right body for the right glaze.

The blue glazes

These glazes are of the same type as the white ones but with slightly lower lead contents. These glazes are characterized by the presence of significant amounts of cobalt (average % Co=0.88) which is responsible for their deep blue colours.

Cobalt was known since long time for its beautiful deep blue colour. It has been detected in some Egyptian glasses dated back to the 15th century BC (Kleinman, 1990). Cobalt had been used by Islamic potters to produce the blue on white wares supposedly in order to produce local imitations of the T'ang ceramics which were ably done.

Since only CoO₄-complex is responsible for the blue colour in glaze, cobalt ores or minerals must have been specially prepared and converted into an oxide or a silicate (Klinman, 1990). The blue glaze contain nickel and significant amounts of iron while the white glaze contains no nickel and lower percentages of iron. Nickel and iron must have come with the cobalt compound. Therefore it can be suggested that a Co-Ni-Fe ore (Linneite) had been worked in such a way that it became completely oxidized during a molten state. Obviously the melt was quenched to obtain a granulate, which was recalcined with a glaze frit. This process is similar to the one described by abu'l-Qasim (Allan, 1973) to make a pigment for a white glaze.

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