

## MULTI – ANALYTICAL CHARACTERIZATION OF XVII CENTURY MUGHAL GLAZE TILES FROM NORTHERN INDIA

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### **Abstract**

*Glazed tile art work is a technique that has been employed for decoration in most of the Mughal monuments during 16 – 17th Century in India. Dakhni Sarai is one of the finest and best preserved specimens of Mughal caravan sarais from India. Glaze tile samples of Dakhni Sarai were analyzed for glaze segments and colours that were used during the Mughal era. The samples were studied using stereomicroscopic analysis, EMPA – WDS, SEM - EDX and by thermal analysis using DTA/TGA. The EMPA-WDS results indicated the usage of lead colorants – tin for yellow glazes, copper - cobalt for blue glazes, and a mixture of two for green glaze tiles. The thickness and slip fusion of the tiles with the inner have also been studied. The result showed that oxides of copper/cobalt/lead used as colorant in varied proportion have imparted color to the glaze under different firing environment and temperature. The presence of copper/cobalt could be either due to its deliberate addition as part of the manufacturing process or as traces in raw material. The results proved that the manufacturing techniques of 16th century Mughal glaze tile art work show resemblance to contemporary Persian tile works.*

**Keywords:** *Glazed tile; Colorant; Vitrification; Quartzite; Sillimanite; Firing environment; EPMA.*

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### **Introduction**

India possesses built cultural heritage from the Stone Age to till date that reflect the architecture, tools and materials used in that era. The fascination in ceramics lies in the possibility of creating beautiful effects with glaze. Glazed tile work was introduced in India with the spread of Islam and its art and architecture and numerous monuments established with tiles works can be found across the country. Besides, highly rich remnants of Persian and Timurid technology of tile making are still preserved in India, that were evidently lost during massive wars and other political problems in Iran and neighboring countries [1, 2]. The workers and their technology were honored by the contemporary Mughal emperors of 16<sup>th</sup> century who called for the Persian artists and asked them to execute glazed tile work in historical buildings [3]. The glazed tile decoration technique emerged as a unique art of its own kind during the Mughal period (1526 – 1857 A.D.) in this sub-continent and it became one of the key ornamental techniques for the monumental buildings of that particular era.

In India, glaze ware came along with medieval invaders from west Asia as earliest glazed tile decoration dates back to 12-13 Century A.D. The medieval Indian potters used both

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the transparent glaze and opaque glaze to decorate the ware. On the basis of their melting point, the glazed materials used during medieval period may be categorized as low, medium and high temperature glaze. Zinc and antimony were low temperature glaze materials as they melt below 1000°C whereas titanium and zirconium oxides were high temperature glaze materials melting beyond 1250°C. Important fluxes used in Indian glazed pottery to help the ingredients to melt were borax, lead oxide, lime and soda potash. In the medieval period, the closed firing pits were a major technological innovation in glazed tile works as they facilitated controlled firing temperature to create reducing and oxidizing environment along with maximum utilization of temperature. The metallic oxides were introduced in this period to obtain different colors in glazed finish. The ceramists produced brilliant colors in many hues by varying firing temperature and environment to obtain entirely different color with the same glaze.

Dakhni Sarai (Fig.1) is one of the finest and best preserved specimens of Mughal caravan Sarais built along the old Highway in the Village Dakhni of Jalandar, Punjab, India. It is said to have been built by Ali Mardan Khan during the reign of Shah Jahan about 1640 A.D. It comprises of one hundred and twenty four cells around a closed quadrangle with two imposing gateway in the centre of the eastern and western quadrangle. Inside the quadrangle are a mosque and a well. The half dome portal of Sarai is decorated with glazed tiles while its interior contains painted motifs over lime plaster. The wall closing the Sarai is strengthened by circular bastion on the exterior at the four corners. The three-storied gateway facade shows recesses and openings on either side, the smaller one being closed with finely pierced tracery screens in red sandstone. The gateway projecting out of the wall is strengthened with octagonal towers, crowned with domical cupolas. The arch spandrels and panels framing the central arch and side openings are decorated with glazed tile-work showing geometrical and floral designs. The whole surface is embellished with arabesque designs executed in yellow, orange, green, turquoise, indigo and white glazed tiles. (Fig. 2) The domed bastions also had similar decorations of which traces remain. According to the Persian inscription above the western gate, the Sarai (Nur-Sara) was built under the order of Nur Jahan Begum. The work started in 1028 A.H. (1618-1619 A.D.) and was completed in 1620-1621 A.D. for the first phase.



Fig. 1. General View of Dakhni Sarai, Jalandar, Punjab, India

The material compositions of Islamic glazed pottery have relatively been well studied [3] and inferences drawn and extended to the tile work, owing to similarities in composition and manufacturing technology. Glazed pottery and tile works of Turkey, Iran and Central Asia have

been well researched. However, in India, the glazed tile has been less studied [4, 5]. Few researches have been carried out hitherto and modest information is available with respect to the material composition of these tiles. The particular decorative technique of glazed tile art work needs to be restored from deterioration. The lack of knowledge on the materials and methodology of the glazed tile art work leads to this interesting and constructive area of research.



**Fig. 2.** Close view of decorative art work of glazed tiles, glaze tile samples and photomicrograph image of blue glaze tile

In India, identification of coloring agent of northern black polish ware (NBPW) and its characteristic glow has been the subject of many investigation and assumptions. *K.B.M. Sanaullah, A. Gosh and K.C. Panigrahi* [4] reported that NBPW black coating has about 13% FeO, which is responsible for black shade. They suggested ferrous sulphate is responsible for coloring and further reported carbon and tarry matters in the pores as being responsible for the enhanced black color [4]. *B.B. Lal* [5] assumed that the NBPW black color is due to some sort of post firing treatment, wherein kiln hot pottery was coated with some organic liquid of vegetable or animal origin. However, the exact nature of firing and nature of proposed ferruginous material used in the manufacturing process were not examined. *K.T.M. Hedges* [6] suggested that the origin of shining black slip lies in the thin layer of magnetic oxides,  $Fe_3O_4$  applied on baked clay which is responsible for its black color. *H.C. Bhardwaj* [7] analyzed the slip and observed that the brilliant color is caused by the application of refined clay and organic liquid during firing, in agreement to *B.B. Lal* [5] suggestions. Further, through a comparative study with the use of binocular microscope and scanning Electron microscope, *D. Sharmin and F. Okada* [8] proposed the application of two kinds of slip on NBPW to create glaze. However, the precise nature of the surface layer still remains unresolved. The detailed manufacturing

technology of these Mughal glazes is still under investigation to envisage the factors involved in improving the quality of glazes. The historic evidence further supports the import of ceramic glazed tile technology from Timurid central Asia during the Mughal period [9 – 11]. Further, *A. Fluegel* [12] has developed a temperature calculator for soda-lime-silica glasses. The same was used to calculate the approximate temperature used for the manufacturing of Mughal glazes. He obtained the melting temperature ranging from 850 to 950°C which is based on silica, soda, alumina, lime, magnesia and potash values without including the colourants amounts. The calculated temperature range for glaze matches the range given in historical reports [13, 14] that has probably been used in the manufacturing of Mughal glazed tiles.

Methodical investigations of the colour glazes of various periods and different places have been carried out and reported globally [15-17]. Yet, there is a lack of scientific research and methodological approach on the glazed tile art work of Mughal period. The main aims of this investigation are to know the composition and nature of the body of tile (base material), the tile manufacturing mode including the baking/firing temperature and the kind of thermo-mechanical changes that have occurred during its manufacturing. The addition of special component to improve the quality of glaze is also a subject of this investigation. For this, geochemical characterization of archaeological pottery played a major role in knowing the manufacturing technique of glazed ware. The aim of this communication is to provide an insight into the manufacturing technology; especially the surface phenomenon and firing temperature and its environment utilized in the 17<sup>th</sup> century Mughal monument, Dakhni Sarai of northern India.

## Materials and Methods

The quantitative determinations of glazes have sometimes been questioned as the material is impure and primitive potters had no means of accurate measurements of the components of the mixture. However, this does not lead us to believe that significant quantitative changes did not occur in glaze manufacturing. The issue of special interest in glazed works is the ratio and other properties of glaze. Analytical results show a distinct and consistent increase in the percentage of lead in later Indian glazes which accounts for greater fluidity [17].

Microscopic examination of the glaze shows the geniality of the glass and amount of un-dissolved pigment present in it [18]. A thin section of the glaze with the body (base material) reflect distribution of different phases of the glass and its inclusion (un-dissolved constituents bubbles etc), the extent of reaction of clay with glaze and penetration of glaze into the body. The microscopic examination also reveals the relation of pigment to glass and is a very important tool to investigate interesting development in the history of glazed works.

Quantitative spectroscopic analysis of the glazes show the difference in trace elements that might give some clue about the sources of ores in relation to the centre of production and extent to which production was controlled by the people in the vicinity of deposits [19]. The ore composition of each deposit must have a unique pattern of trace elements and elements which are essential for the recognition of those patterns must not be subjected to volatilization during firing. However, volatilization of the constituents in firing would cause only minor change if the atmosphere is oxidizing [20]. The examination of polished surfaces of glazed tiles using SEM with attached analytical facilities is a powerful technique for surface analysis [21]. SEM attached with EDX provides a rapid and excellent method to study voids, inclusions and examination of fractured section [22].

As the monument is of national importance and it is a built cultural heritage, no direct sampling was carried out. Samples are being limited to portions of tiles that had fallen off the building and were sourced from of the monument. A total number of seven glazed tiles (green,

yellow and blue coloured samples) (Fig. 2), that had tile body and its overlying glaze layer were examined.

The glazed samples were thoroughly washed with cotton swabs and distilled water until they were free from any adhering accretions. Subsequently, the samples were dried at about 80°C overnight and polished vertically for examination under microscope. The samples were observed under Cole-Parmer Stereo zoom camera microscope with built-in 2.0 mega pixel digital camera at different magnification and photo micrographic images were also taken. The chemical composition of the base material of the glazed tiles was determined by X – ray florescence spectroscopy (Phillips PW 1410, Holland) as per standard procedure by mounting the compressed pellet in the sample holder of XRF set up. High resolution scanning electron microscope attached with EDX make of FEI Quanta 200 was used for elemental mapping of glazes as well as the adhesive sip materials. The SEM images were recorded in various magnifications with the beam intensity of 20kV. Differential thermal analysis (DTA) and thermal gravimetric analysis (TGA) were carried out using standard procedure with alumina as reference with Perkin-Elmer (Pyris Diamond) instrument for all the three glazes as well as the base material.

JEOL Electron Microprobe Analyzer JXA 8200 equipped with a secondary electron (SE) and a back scattered electron (BSE) - detector, wavelength dispersive X-ray spectrometers (WDS) were used to analyze the specific areas of the cross-sectioned samples. The voltage range of the energy dispersive X-ray spectrometer (EDS) was from 0.2 to 40kV and the current range was from  $10^{-12}$  to  $10^{-5}$ . This allowed an accurate and quantitative analysis of the glaze layer.

## Results

### *Chemical analysis of Glazes*

The chemical composition of the seven samples of glaze tiles was determined with Electron Microprobe Analyzer and the results are shown in the Table 1.

**Table. 1** Chemical Composition of Glazed tile samples determined by Electron Microprobe Analysis (EPMA)

Sample Colour	Sample No	Na <sub>2</sub> O	CaO	K <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	CuO	CoO	PbO	ZnO	P <sub>2</sub> O <sub>5</sub>
Blue	1	18.2	2.71	1.72	1.15	6.1	1.45	64.5	0.32	0.08	0.18	-	-	0.21
Blue	2	15.4	1.9	2.45	1.08	6.5	1.65	68.3	0.38	0.07	0.16	-	-	0.25
Yellow	3	14.5	2.02	1.7	1.45	3.85	0.85	55.35	0.21	1.1	-	15.85	0.26	0.24
Yellow	4	15.2	1.45	1.98	0.85	4.1	1.44	57.45	0.34	0.8	-	14.35	0.32	0.15
Green	5	14.62	1.57	1.57	0.72	6.2	1.52	56.7	0.34	1.27	-	11.25	0.32	0.15
Green	6	17.2	1.46	1.69	0.58	6.31	1.45	54.3	0.3	1.78	-	9.52	0.25	0.08
Base	7	1.86	0.96	0.69	0.4	0.85	0.79	96.05	0.17	-	-	-	-	0.07

From the above analytical data, it is observed that in the yellow glaze tiles, lead in association with tin was used with inclusion of traces of zinc. An average 0.2% zinc oxide noticed in the samples probably comes from impurities of the lead oxide. The EPMA analysis (Table 1) also revealed the presence of cobalt and copper in blue glazes. Historically cobalt was used in glazes to give an enriched blue color [23 – 26]. Addition of copper in similar amounts (0.2– 0.5%) to cobalt was reported to impart blue colour in some of the Indian glazes [3]. The presence of lead, tin and zinc in identical ratio as in the yellow glazes (Table 1) suggests that the green glazes were made up in part from similar constituents as the yellow glazes [3]. The chemical composition of the base material for the yellow glaze was also determined using



EPMA – WDS methods. The result shows that the base material is primarily a quartzite having a silica percentage up to 96% with small amount of sodium and calcium oxides as impurities.

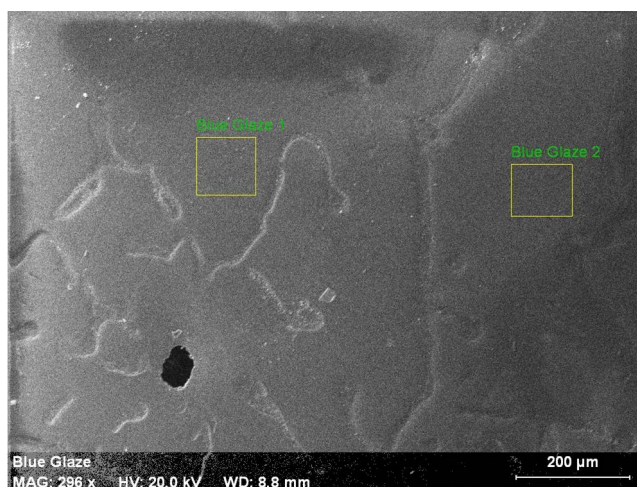
#### ***Micro structural investigation***

The glazed tile samples (one sample for blue, yellow and green) were investigated under visual examination. All three tile fragments were identical, encompassing dirt covering brown coloured bodies with an overlying glaze layer. The glazed tiles examined have a general consistency in thickness of about 1-2cm. In all three glazed bodies, interface is visually apparent as a narrow whitish colour bands just below the glazed layers. All three glazed samples were examined for their microstructure under stereomicroscope under a magnification of 0.2 to 0.7X and photomicrographs of the image were also recorded. The result of investigation through photomicrograph and SEM EDX are as under:

#### ***Blue glazes***

The micrograph of blue glaze showed the existence of dirty white base material for the glazed ware. The inorganic adhesive as slip material is observed between the base and glazed work that has also been fired along with the glaze. The slightly reddish orange tinge also detected in between the base and the glaze may be due to the high temperature firing and contribution on account of molecular changes of ingredient materials. The white particles observed in the micrograph are probably circular quartz grains. As the quartz grains are generally crystalline, the circular shape they have acquired is on account of edges dissolution during firing. The glaze is very thin under surface as is clearly visible under the microscope. The unequal shade of blue color observed in photomicrographs, may be due to uneven thermal impact and unequal dissolution of coloring elements.

The blue glaze was examined using a scanning electron microscope under a 296X magnification. SEM photomicrographs of the blue glaze (Figure 3) show the presence of fine, smooth and amorphous surfaces with very fine streak lines. The two segments of blue glaze measuring about 150  $\mu\text{m}$  of length were selected for EDX analysis. The peaks were recorded and the result quantified in the form of oxides through the EDX. The tinge of copper oxide and cobalt oxide are evident in the analysis probably used as coloring agent. The presence of copper/cobalt could be either due to its deliberate addition as part of the manufacturing process or as traces in raw material [23, 24]. The varied composition of ingredient and firing in highly oxidizing environment as observed through EDX analysis might have lead to the development of blue color of glazed tiles [27 – 29]. The second segment observed under EDX also reflects similar result confirming the surfaces composition.



**Fig. 3.** SEM image of blue glaze tile

### Green glazes

A better uniformity of coloring agent is inferred in green glaze under photomicrograph and the glaze layer observed is also thicker than the other two glazed types (Figure 4). The vertical section of the glaze shows very fine priming layer of white color that perhaps is the inorganic adhesive slip layer. Probably, the component ingredient of green glazed material required more roughness and binding to the base surface and to match the coefficient of expansion, red ochre particles have been incorporated in the base as observed through photomicrograph. The yellow and blue glaze bases probably do not have this requirement.

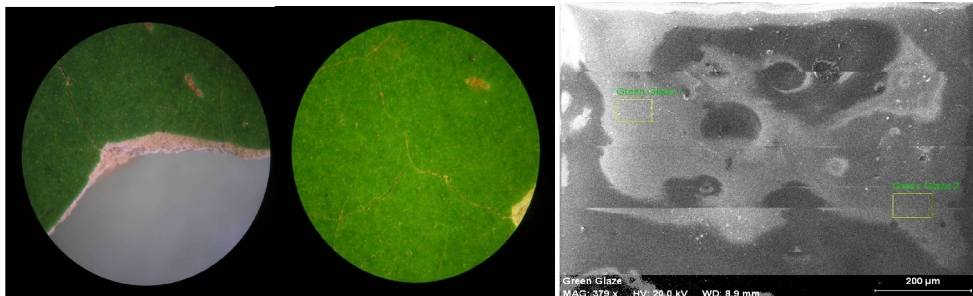


Fig. 4. Photomicrograph image (a) of green glaze tile and SEM image (b)

The green glaze surface is thicker than other two glaze types as observed through stereomicroscopic images. The superior thickness of the green glaze was also confirmed by SEM micrograph image (Fig. 4b). The SEM micrograph of green glaze recorded at 379X magnification reveals the development of uneven surface thickness due to application of thick layer glazed material before firing. The peaks under EDX were again recorded at two different areas of about 100 µm length. The EDX data shows high inclusions of copper oxide up to 3.5% as coloring agent in association with traces of zinc and higher quantity of lead oxide as coloring agents, Table 2. The changes in the firing environment and difference in copper percentage have helped in the development of thicker green color. Identical results were observed in the second targeted segment.

### Yellow glazes

The yellow glaze under microphotograph seems to be more compact than the other two types with characteristic priming layer between base and glaze. The vertical section shows the thickness of the base and top glazed layer (Fig. 5). In the base of yellow glazed tiles, traces of red ochre inclusion are observed along with quartz.

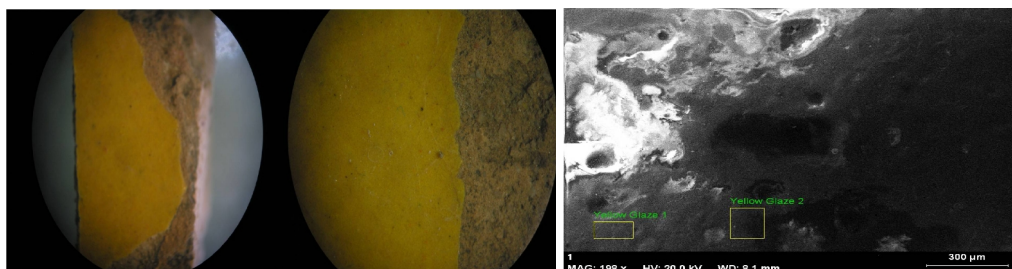


Fig. 5. Photomicrograph image (a) of yellow glaze tile and its SEM image (b)

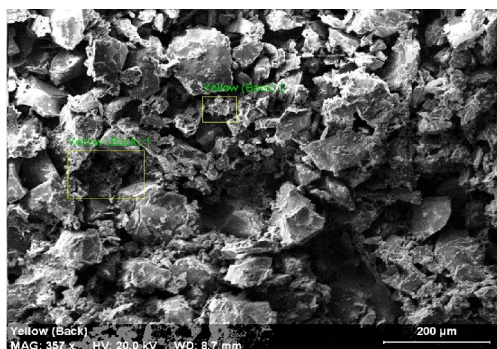
**Table 2.** Spectrum data obtained from SEM – EDX

Element	Series	Unn. C [wt.-%]	Norm. C [wt.-%]	Atom. C [at.-%]	Oxide	Oxide C [wt.-%]
<b>Spectrum : Blue</b>						
Aluminium	K-series	16.04	16.04	10.41	Al <sub>2</sub> O <sub>3</sub>	77.23
Silicon	K-series	3.75	3.75	2.34	SiO <sub>2</sub>	20.45
Cobalt	K-series	0.73	0.73	0.20	CoO	2.32
Oxygen	K-series	79.49	79.49	87.05	O	154.88
Total:		101.37	100.00	100.00		
<b>Spectrum : Green</b>						
Aluminium	K-series	37.80	35.1	25.43	Al <sub>2</sub> O <sub>3</sub>	76.64
Lead	K-series	4.97	4.75	3.63	PbO	13.19
Silicon	K-series	1.83	1.93	1.35	SiO <sub>2</sub>	5.01
Copper	K-series	2.08	2.19	0.68	CuO	3.34
Oxygen	K-series	53.15	56.03	68.91	O	21.72
Total:		99.83	100.00	100.00		
<b>Spectrum : Yellow</b>						
Aluminium	K-series	27.66	27.68	18.76	Al <sub>2</sub> O <sub>3</sub>	78.64
Lead	K-series	4.97	4.92	3.97	PbO	14.19
Silicon	K-series	1.99	1.94	1.30	SiO <sub>2</sub>	6.19
Copper	K-series	1.14	1.12	0.33	CuO	2.08
Oxygen	K-series	65.91	64.34	75.64	O	48.92
Total:		101.67	100.00	100.00		
<b>Spectrum : Yellow (base)</b>						
Aluminium	K-series	3.40	3.40	2.20	Al <sub>2</sub> O <sub>3</sub>	15.56
Silicon	K-series	16.31	16.31	10.14	SiO <sub>2</sub>	84.44
Oxygen	K-series	80.29	80.29	87.66	O	142.03
Total:		100.00	100.00	100.00		

The yellow glaze surface was observed through scanning electron microscope under a magnification of 198X and a beam intensity of 20kV. Microscopically, the glaze surface seems to be amorphous with very small pits in between (Fig. 5). Two segments of about 150µm were selected and energy dispersive analysis carried out. The peaks were identified and components of glaze were quantified as oxides as shown in Table 2. From the composition it is observed that the glaze portion is primarily composed of 78% alumina, 14% lead oxide, 6% silica oxide and copper oxide 2%, added to obtain desired color during firing. The firing was done in reducing atmosphere that helped lead/copper oxide to change color in the presence of alumina and silica. The second targeted segment has also shown equivalent observation.

#### *Intermediate layer (Slip layer) analysis*

Scanning electron microscopic images of the intermediate adhesive layer were also taken at a magnification of 357X. The SEM photomicrograph (Figure 6), clearly illustrated a fine layer of adhesive embedded between the quartz and alumina grains in the form of small and uniform particles.

**Fig.6** SEM image of Yellow base material



The bigger size crystalline grains resemble the base material i.e. quartz and alumina. The nature of adhesive layer and its composition was determined by carrying out EDX analysis on two different segments and the result is shown in Table 2. On the basis of this data it is observed that slip layer mostly consists of 85% silica, around 15% alumina with plenty of oxygen in it.

**Thermal Analysis**

*Blue glaze*

The blue glaze sample was subjected to thermal analysis using DTA/TGA to understand the firing changes and the result of the analysis is shown in Figure 7a. From DTG graph it is evident that at 208°C the absorbed water has escaped from the material interiors. At 1220°C the vitrification process has started and the secondary mullite crystallized out of liquid phase. Beyond 1400°C it was not possible to make any measurement due to instrument limitation. The TGA graph shows the endothermic changes at a rate 176 mJ/mg between 1100°C and 1300°C.

*Green glaze*

The green glaze sample was observed under DTA/TGA and the result is shown in Figure 7b. From the DTG graph it is observed that the firing change has occurred at 977°C probably due to formation of trydymite and around 1100°C the free silica has started converting into crystallobalite. The TGA graph shows various endothermic and exothermic changes between 900°C and 1250°C.

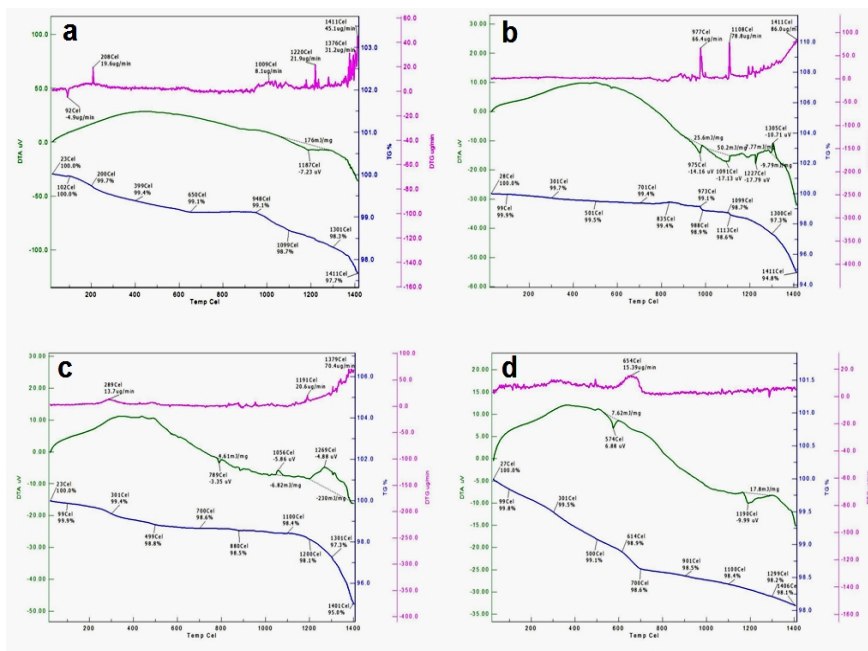


Fig. 7. TG-DTA curves of the samples: a – blue glaze; b – green glaze; c – yellow glaze; d - yellow base material sample

*Yellow glaze*

From the TG-DTA graph, Figure 7c, for the yellow glaze it is observed that the process of vitrification has started at about 1200°C. The sample is showing transformation from 1190°C onwards at a rate of 20.6 μg/min due to phase transition of sillimanite and later at about 1400°C firing transition was observed and the remaining amorphous silica has started converting into cristobalite. Firing changes beyond 1400°C could not be measured due to equipment limitation.

The TG graph shows weight reduction of up to 95% with exothermic changes beyond 1056°C. At around 300°C firing phase transformation was observed where in alpha cristobalite has changed to beta cristobalite.

#### *Yellow base material*

The yellow base material previously analyzed with EMPA, Table-1, was also observed under DTA/TGA to know its firing details, Figure 7d. The EMPA analysis denotes that the base material is mainly composed of more than 96% of silica whose firing change cannot be determined on the available equipment. Therefore, no DTG change was observed except slight changes at 650°C due to crystallization water loss. The endothermic changes were observed at about 1200°C, probably due to process of vitrification and crystallization of the secondary mullite.

## **Discussion**

The art of glazing has been reported from Mohanjo-Daro [30]. In Indian sub-continent traces of glazing on ceramics has also been reported from Harrapan site [31] though distinctive pottery of Harrapan civilization is mostly Harrapan red ware [32]. The ceramists calculate the composition of glazes in terms of proportions of basic, intermediate and acid oxides corresponding to network modifiers, intermediate glass formers and network formers, respectively. Since space in the glass network doesn't need be filled, the glass does not have fixed proportions of components. As the proportions of component vary, the properties of glasses are modified. However, the low melting glazes are of special interest to archaeologists because they must have been first to be discovered. The oxides of metals as chief colorant played a very important role in glaze works. As the color of the glaze is influenced by the state of metallic oxides, its position in the network and the kind of atoms surrounding it, the same metallic elements may have more than one effect [33]. Therefore, the ceramists must be aware how the various components of the glaze will impact its shrinkage and elasticity and the problem of avoiding unequal shrinkage is not entirely matching the co-efficient of expansion. Other defects that must be controlled in manufacturing glazed tiles are blistering due to formation of gases on trapped air, incomplete dissolution of the constituents, re-crystallization (devitrification of glass) and extremes of viscosity [34].

The lead-tin yellow pigments have been widely studied [35, 36] and generally two kinds were defined: type I ( $\text{PbSnO}_4$ ), and type II ( $\text{PbSn}_{1-x}\text{Si}_x\text{O}_3$ ). Both were used as paint pigments in Europe before the 18th century but the type II was mainly associated with the manufacturing of glass and glazes [35- 36]. We could not ascertain the use of lead-tin yellow type I rather than type II. In fact, type II is obtained by heating type I at around 950 °C ( $\text{Pb}_2\text{SnO}_4 + \text{SiO}_2 \rightarrow \text{PbSn}_{1-x}\text{Si}_x\text{O}_3$ , at  $T = 800\text{--}950$  °C) [35-36] which is the same range of temperatures used for fusing glazes during the Mughal period as documented in ancient texts [13-14]. It is possible to say that the green glaze was obtained from a mixture of lead and tin-containing raw materials (mixed in the same proportion as for the yellow glaze); cobalt and copper as blue colouring agents.

The green, yellow and blue glazes are showing contents of copper/cobalt/lead/tin as colouring material. The presence of copper/cobalt could be either due to its deliberate addition as part of the manufacturing process or as traces in raw material [4-5]. The varied composition of ingredient and firing in highly oxidizing environment as observed through EDX analysis, might have lead to the development of blue color of glazes. The change in the firing environment and the difference in copper/lead percentage have helped in the development of thicker green color whereas in case of yellow glazes, the firing was done in reduced atmosphere that helped copper/ lead oxide to change color in the presence of alumina and silica. The yellow base material is primarily a quartzite having high silica percentage.

In blue glazes, at 1220°C the vitrification process occurred and secondary mullite crystallized out of liquid phase. The firing change occurred at 977°C for green glazes probably due to formation of trydymite and around 1100°C the formation of crystallobalite has also taken place. The phase transition of sillimanite from 1190°C and later at about 1400°C firing transition was observed in case of yellow glaze, conversion of silica into cristobalite has also occurred. The presence of copper in blue and green tile samples could be the deliberate addition during manufacture or as from raw materials.

## Conclusion

The present investigation provides an insight into manufacturing technology and surface gloss/firing process employed in glazed works of 17<sup>th</sup> century Mughal monument using stereomicroscopic analysis, SEM with EDX and by thermal analysis using DTA/TGA. The EMPA - WDS determined the chemical composition of the glazes that shows the possibility of manufacturing of colored glazes. It confirms that the usage of colorants of lead – tin for yellow glazes, copper and cobalt for blue glazes whereas mixture of two (yellow glaze and blue glaze) for green glaze tiles. The thickness and fusion of slip with the inner core along with the minerals present have also been studied. The result shows that copper/cobalt/lead and tin oxide were used as colorants in various proportions that have imparted color to glaze under different environment of firing and temperature. The presence of copper/cobalt could be either due to its deliberate addition as part of the manufacturing process or as traces of raw material. The results prove that the manufacturing technique of 17th century Mughal glaze tile art work has resemblances to Persian tile works.

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