INTERNATIONAL JOURNAL OF CONSERVATION SCIENCE

ISSN: 2067-533X Volume 7, Issue 4, October-December 2016: 1065-1070 www.ijcs.uaic.ro

SEM-EDX MICROSTRUCTURE CHARACTERIZATION OF PTOLEMAIC FAIENCE BEADS FROM EGYPT

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Abstract

In this study faience beads from Saqqara, Egypt, monochrome and with a diameter of 2-3 mm, dating from the Greco-Roman period, were analyzed by SEM-EDX. SEM was employed in three modes depending on the obtained data; imaging, composition analysis and elemental mapping. The results show that the faience beads were modeled manually by hand, as well as that plant ashes was used as alkaline flux agent rather than natron. Using the elemental mapping playing important role in determination of glazing technique, where cementation is the used technique in studied samples.

Keywords: *Beads; Faience; Cementation; SEM-EDX; Elemental mapping*

Introduction

Beads are an ornamentation form created by people; in ancient Egypt, women and men used them as necklaces, amulets collars, pendants, anklets, bracelets and girdles. In excavation sites, beads exist because they were used by all society classes [1]. Faience manufacture started in the fourth millennium B.C. [2, 3]. The body of ancient Egyptian faience comprises quartz mixed and water added to the mixture, alongside an organic binder that may have been added in order to shape the desired objects [4]. A glaze layer used to cover the faience body, specifically a mixture of quartz, alkali flux or powder of calcium carbonate besides a source of color charged the glaze mixture on the top of the body paste to form a molten glaze layer [5, 6].

Three techniques have been proposed for shaping Egyptian faience. The first is a modeling technique that was used in the pre-dynastic period, in which the objects are modeled by hand. The second is molding, faience being shaped using a rigid frame, and the third technique is abrasion, which combines the two previous techniques [7, 8]. Three techniques of glaze application were used to form glaze a layer on the faience body: application, efflorescence and cementation. The application technique is based on mixing small particles of silica, lime and alkalis with water to form slurry [9]. The mixture was applied directly over the body, or the object was dipped in it, creating a powder coating that transforms into glaze during firing; evidence of this technique comes in the form of brush marks, drips and glaze running lines [10, 11]. The second technique is efflorescence, in which alkali salts are mixed with particles of quartz, and after the evaporation of the water the alkali migrate from the core to the surface. A salt crystalline network and a thin layer of glaze formed when the object was fired [12, 13].

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The third technique is cementation, in which the object is formed, dried and buried in the glaze compositions and a thin glaze layer forms upon burning. The similarity between the techniques used in beads production makes the detection of each difficult by means of traditional analytical instruments. In this work, SEM-EDX was used to examine the microstructure and chemical compositions of faience beads from the late period of Egypt. The work aims to apply elemental mapping to outline the aspects of small items of beads manufactured during the Ptolemaic period.

Materials and methods

Samples

The samples were collected from a wooden coffin assigned to the late Greco-Roman period in Egypt, found in the Maya tomb. The samples are pale turquoise and their diameter is 2–3 mm.

ESEM and micro X-ray analysis

The chemical compositions and microstructures of the beads was determined using Environmental Scanning Electron Microscope (ESEM). A Philips XL-30 ESEM was used in this study, the X-ray microanalysis was carried out using an EDX detector, and the Energy Dispersive Spectrometry (EDX) data acquisition using a GENESIS 6.X software. A backscatter detector used to give excellent atomic number contrast. ESEM provides three types of data about morphological and chemical compositions of the beads: imaging, analysis and elemental mapping.

Results and discussion

Imaging

Scanning Electron Microscope (SEM) results of the studied beads microstructure indicate that quartz grains in the body appear in an angular shape, which shows the raw sand was ground before it was added to the mixture. Quartz grains size is 10–40μm; varieties in the quartz sizes cause heterogeneity of the bead's matrix. The presence of air bubbles originated in the crushed body matrix in size 30–60μm; these air bubbles were trapped during the preparation of the matrix raw materials (Fig. 1), and are weak points of the beads body. The bead's matrix is non-glassy structure.

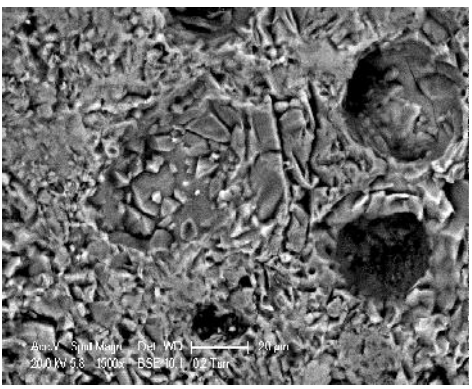

Fig.1. SEM image of bead's body shows the difference in quartz grains size and pores inside, 1500X.

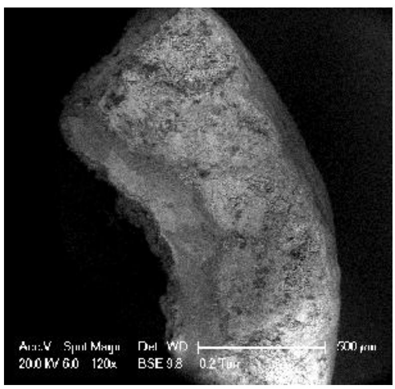

Fig. 2. SEM image of bead surface shows heterogeneity of bead's surface confirms beads modeled by hand, 120X.

The SEM images samples show the manufacturing technique assumed its shape modeled by hand and then a hole drilled to create faience beads (Fig. 2).

Interaction zone between the glaze and the body is rare in the studied samples since the glaze is well defined and the body clears where it finishes and the glaze where it starts (Fig. 3). Meanwhile, there is parts in the same samples include interaction zone, it is difficult to identify the sharp line between the crushed quartz body and the glaze. The disappearance of the interaction zone shows that two firing steps carried out, firing of the body and then glaze burned (Fig. 4). The glaze thickness is 40–50μm and also presents cracks and pores as result of degradation of the beads (Fig. 5). Weathering of the glaze is observed, where the glaze appears pale and cracked under the microscope.

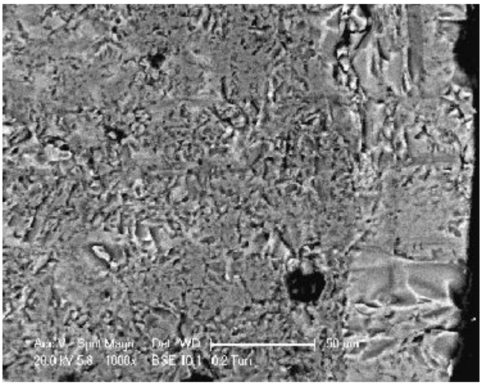

Fig. 3. SEM image of bead's cross-section indicates no interaction zone between body and glaze, 1000X.

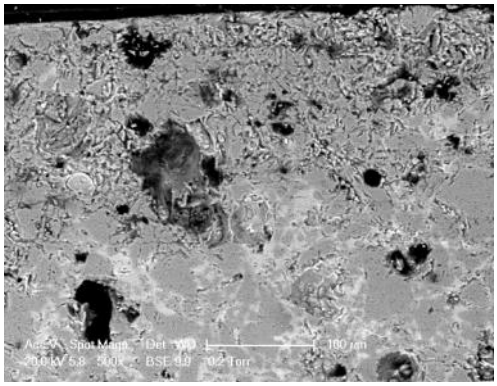

Fig. 4. SEM image shows interaction zone between the beads body and glaze, 500X.

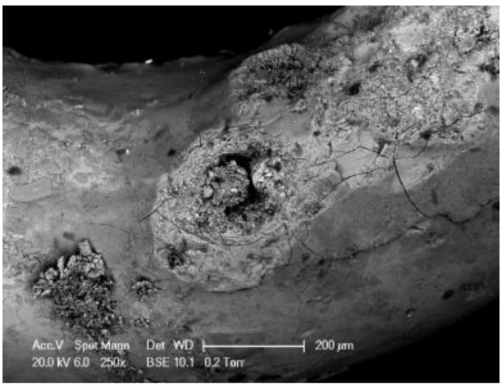

Fig. 5. SEM image shows crack and damage in the glaze layer, 250X.

Beads analysis

The chemical compositions of body and glaze as analyzed by SEM-EDX show that the body mixture consists mainly of quartz (SiO_2) in concentration 80–95%. Microanalysis of the matrix indicates the presence of the additional trace elements Na, K, Al, Fe and Ca. Alumina Al_2O_3 presents in the body due to the presence of clay impurities in the body mixture. The analysis of the body shows that elements of alkali salts were added to the raw materials during bead's production. However, salts caused partially fuse of the body compositions and hardening beads after burning. The presence of Cu, Na and K in the body shows that glaze mixture was intentionally added to the body mixture for hardening the beads and lowering the firing temperature.

Glaze analysis was conducted on the area where only glaze is present; areas with cracks and missing glaze were avoided. The incomplete melting of the glaze components was caused when a low amount of alkaline was present in the glaze compositions. Microanalysis of the glaze and body shows that K and Na are present in the body and the glaze. The potash and soda contents in the glaze are 0.5–1.4% K and, respectively, 0.3–1.5% Na; however, the potash and soda contents were higher than it was expected for natron. Furthermore, the presence of Na and K in the results reveals that the use of plant ashes instead of natron was common in ancient Egypt. Soda-rich plant ashes used in the near east and Egypt in the production of faience [14, 15]. All the studied samples are colored using copper (1–3% CuO).

Beads mapping

The elemental mapping of the surface shows the distribution of the chemical compositions, homogeneity, and heterogeneity of the analyzed surface [16]. Maps of the beads body and glaze show that small amounts of the glaze mixture added to the bulk components that can enrich the cohesive between the glaze and the underglaze matrix. The amount added to the matrix was not enough to form efflorescence glaze. Sample's maps show that copper is present throughout the body mixture and dissolved in the glaze layer as a colorant. In the studied samples, SEM-EDX maps show that glaze formed in thin layers associated with the matrix, and no interaction zone formed (Fig. 6).

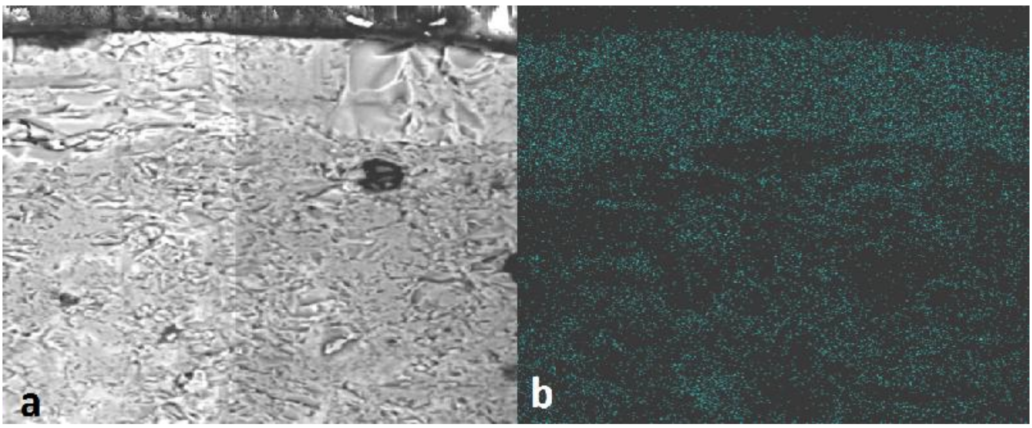

Fig. 6. SEM image of faience bead (a) body and glaze with no interaction zone (b) elemental map of Cu shows the sharpness of glaze on the bead by cementation technique.

In parts of the samples the glaze penetrated into the body from outside and vice-versa, the glaze grows from inside to outside. The glassy phases in the cores and the thick interaction layers suggests that glaze compositions were mixed with the body mixture prior to forming the faience bodies in order to produce a hard body after firing. In contrast, the absence of interstitial glass phases in the body and the interaction layers suggest that the firing temperature didn't reach the critical degree of fusion. However, in spite of these difficulties in determining the method of glazing, the microstructure of faience remains important in assessing the production technology. Elemental mapping of the samples using SEM-EDX found that Na – copper rich phases and wide dispersed copper content, the studied samples shows in part of the samples the glaze is in a layered structure, the elemental map of Cu and Ca are correlated (Fig. 7).

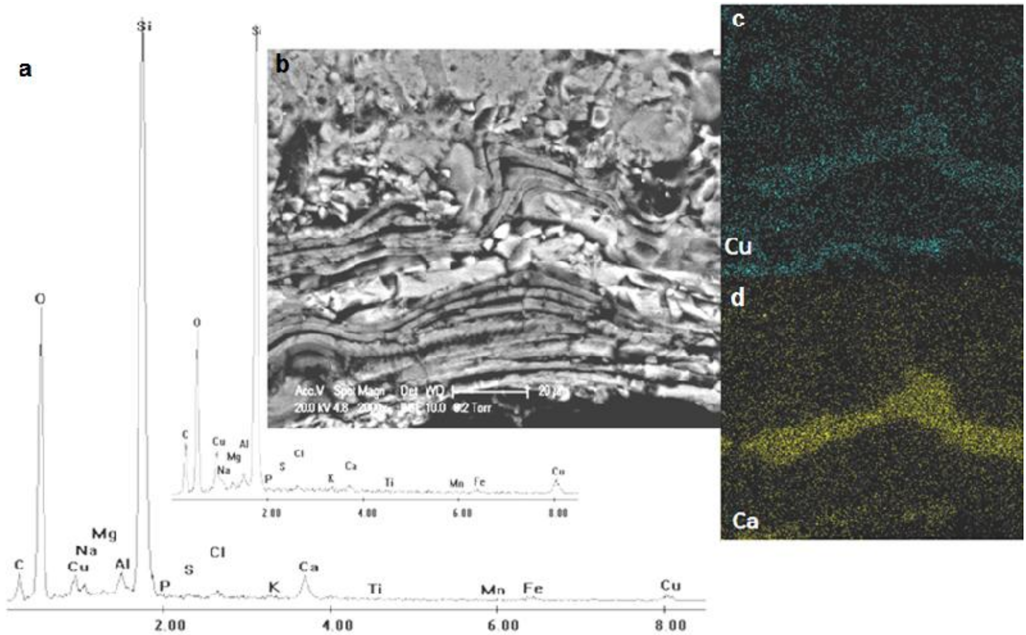

Fig.7. (a) EDX microanalysis of turquoise glaze. (b) Multi-layered structure of the glaze. (c) Elemental mapping of Ca in the layered structure. (d) Elemental mapping of Cu in the layered structure.

Conclusion

The faience beads from the Ptolemaic period in Saqqara were manufactured from crushed quartz; the grain size of the quartz varied and angular edges and some of the glaze mixture was added to the body for hardening the beads during firing. Based on the analytical results of the SEM-EDX, it appears that that plant ashes rich in salts were used as a flux in the production of the Ptolemaic beads from Saqqara, rather than natron as flux agent. The microscopic investigation indicates that beads were modeled by hand and then a hole was drilled into the body. The interaction zone between the body and glaze and elemental maps played a dominant role in the determination of glaze application technique. Since cementation glazing technique was used for the Ptolemaic beads in Egypt, there was no evidence to apply efflorescence technique. Copper was used as a principal component for charging the monochrome beads, with different percentages of concentrations.

Acknowledgements

The author would like to thank Mr. Moustafa A. Abd El Fatah the general director of conservation in Giza for providing the author with samples.

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Received: February, 22, 2016
Accepted: September, 15, 2016