

Original article

RESTORATION OF AN ARCHAEOLOGICAL CERAMIC JAR FROM MAMLUK PERIOD (1250–1517A.C.), AL-FUSTAT, EGYPT: AN ANALYTICAL STUDY

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Abstract

Ceramics were one of the oldest and most important industries that had evolved significantly all over the Islamic periods. Jar is a large ceramic vessel that was used in the Islamic period. It was excavated at Al Fustat, Egypt by the Faculty of Applied Arts, Helwan University and was added to the college museum in 1945. It was found that it dated back to the Mamluk Period (1250–1517A.C.). The jar was in a very poor condition; it was fielded and was restored with unsuitable materials that turned it into several parts, especially at the base. In addition, it suffered from many deteriorations that include cracks, decay and crystallization of salts. Furthermore, many shards were broken from the body and some were missed. The aim of the current study was to study and restore the chemical and mineralogical composition of the clay body, the glaze and the crystallized salts. Different analytical methods were used including; X-ray diffraction (XRD) and Scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS). Chromite (Cr_2O_3) was used to obtain green glazes. Different restoration treatments were carried out on the jar, such as mechanical and chemical cleaning, consolidation, bonding and replacement processes. The results suggested that the salt was made of Halite mineral (Sodium chloride) and Calcite (CaCO_3), while the clay body of the jar was made of ferruginous, highly calcareous clay in an oxidizing atmosphere. Additionally, minerals such as silica, hematite, gehlenite and lime were identified.

Keywords: *Ceramics, Al Fustat, Jar, Mamluk period, Restoration, Reconstruction*

1. Introduction

1.1. The archaeological and historical studies

In this study, a jar of glazed ceramic belonging to the Mamluk period (1250-1517A.C.), and discovered in Al-Fustāt, was supplied from the archaeological excavation collections site. It consisted of a ceramic body that was covered with a green glazed layer. Al Fustat is located between Babylon Fortress and Al Moqqatam Mount. Al-Fustāt is the name of the first Islamic capital of Egypt that was founded shortly after the Islamic Ummayyad

conquest of Egypt in 641A.C. The city's name came from the Arabic word Fusā that meant tent or pavilion. Its location was chosen because of its clear advantages commanding the approach to Upper Egypt and the East-West routes by land and sea [1]. It was the capital of Islamic province of Egypt during the Umayyad and Abbāsīd rules and under succeeding dynasties, until it was captured by the Fātimīd general Jawhar. On the east bank of the Nile

River, to the south of modern Cairo, it grew into a permanent city out of an Arab camp set up for the siege of the Byzantine fortress of Babylon, but it was developed rather chaotically. The Abbāsid governors, thus, chose to reside in a northern suburb, i.e. Al-Askar, while the Tūlūnid dynasty built a new quarter, i.e. Al-Qatāi, to serve as its capital. The city's prosperity, derived from a lively commerce and fine glassware and ceramics industry,

facilitated the survival of Al-Fustāt after 969 A.C. when nearby Cairo became the capital of Fatimid Egypt. In 1168 A.C. the town, which had never been fortified, was destroyed by fire to prevent its capture by Frankish armies. It was rebuilt a few years later by Saladin, who joined it with Cairo. The city's very name was gradually replaced by that of Al-Qāhirah (Cairo), the breadth of the Islamic Mamluk state was showing in fig. (1).



Figure (1) Shows a map is showing the breadth of the Islamic Mamluk state

Al-Fustāt, the center of ceramic production in medieval Egypt, has been haphazardly excavated. Consequently, the opportunity it would have afforded for stratigraphic dating assigning objects dated in accordance with the levels on which they were found was lost. In the Islamic periods, glazed ceramic was produced all over the Middle-East. However, one of its well-known production Centre was Al-Fustāt [2]. It remained the main production center of Egypt. Glazed ceramic was made up of a mixture of natural materials that were combined and formed into shape by a variety of processes and transformed by heat to create a solid material that was not found in nature. Glazes were the coatings of the pottery surface and were silica-based. The fusion of silica at low temperatures (700-800 °C) was produced by adding flux agent such as lead or

alkaline or both. These surface treatments could be applied to the dried pottery before or after the first firing and later subjected to subsequent heat treatment. Glaze charged with coloring agents gave a homogeneous color to ceramic surface and, depending on the presence of pacifiers; the glazed surface would be opaque or transparent. Decoration, also, had a functional aim in protecting or permeabilizing the pot's surface [3]. There were some investigations in the composition of ceramic objects from Al-Fustāt that were carried out on both glaze. The ceramic body, in the current study, was rarely taken into consideration regarding the technical aspects [4]. Compositional date played a key role in solving questions concerning dating and the provenance of ceramic objects. It could, also, provide knowledge about raw

materials and colorant agents used during the body and glaze manufacture and found out details of glaze production techniques and other decoration types. From year to year, the increasing role of archaeometric studies in the investigations of cultural heritage could be observed [5]. Some of them were produced as modular pieces and then joined together. After forming the body and the base, the vase was turned upright and supported by chuckle while the neck and rim were formed and the handles were attached last [6]. The changes that occurred in minerals on heating were of many kinds; both physical and chemical. They included dehydration, oxidation, reduction, inversion, decomposition and fusion. Dehydration occurred in lower temperature that varied and might be accompanied by swelling as water of crystallization was converted to steam. Oxidation and reduction were controlled by firing atmosphere and occurred over a considerable temperature range. There were many changes that might have an important effect on color. Inversion was a physical change in atomic structure that took place at different temperatures with the different minerals. Decomposition

took place over a wide temperature range, depending on composition. Fusion or melting of substances occurred in higher temperature range. We would review the evidence of these changes, grouping them with respect to their effect on the pottery, attempting to cover the major kinds of temper and to consider the nature of these changes [7]. Generally, the colors employed on ceramic objects were usually obtained by metallic oxides because of their high stability in molten silicate glasses [8]. The metallic oxides could be used in a pure state or in a mixture called frit and premelting of the pigment together with fluxes such as Pb, Na and Sn. After cooling down, this mixture was finally grinded and applied over the enamel [9]. Green glazes had different tonality with varied composition. They might comprise two main coloring oxides; copper and cobalt. Copper oxide was the main oxide responsible for the green color. Copper might be prepared by mixing either copper oxide or available Cu carbonate ores such as; malachite and Azurite [10]. The shape of jar is common in this period and the degree of color, fig. (2-a, b, c, d)



Figure (2) Shows **a.** ceramic jar from Al-Rekha Saria, **b.** Irak or Seria, **c.** Iran (9th century) Glazed earthenware, **d.** Possibly Egypt, (9th- 11th century) stone paste: monochrome glazed.

The main aim of the present study was to identify the chemical and the mineralogical composition of the glaze, the clay body of the jar, the crystallized salts and carry out restoration

process. The chemical, mineralogical and structural characterization of ancient Islamic ceramic could shed light on the provenance of raw materials for ceramic production and

determine the technological processes related to ceramic manufacture [11,12]. Different analytical methods were used, including; X-ray diffraction (XRD) and Scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS) to study different samples of crystallized salts, clay body, and glaze layer. Complete restoration processes were carried out on the jar,

1.2. Deterioration phenomena of the jar

The jar was in a very poor condition because it was fielded and restored in the wrong way with unsuitable materials that turned it into several parts, especially at the base. In addition, some parts were missed. There were many deterioration symptoms, including; cracks, decay and crystallization of salts. It was obvious that this jar was subjected to an incompatible restoration process in the past. It crashed and the old restoration where it was joined together by using a layer of gypsum with a large quantity of white adhesive

such as cleaning, bonding of broken parts and replacement of the missing parts. Paroloid B-82 (an acrylic resin that retained the outstanding characteristics of acrylic resins, such as transparency, durability, flexibility, and resistance characteristics. It was supplied as a colorless transparent solution at 50 % solids in toluene) was, also, available, in a solid grade [13,14].

polyvinyl acetate with filling the void with a piece of colored cloth, led to a lack of adhesion in the broken parts in the body. It fell again with increases of adhesive. Furthermore, many shards from the body were broken and some were missed in the body, the neck, and the base. There were, also, cracks in the glaze body and in the clay body. Lamination illustrated the crystallized salt on the surface. The missing parts of jar are in the body, the neck, and the base, the deterioration phenomena of the jar are shown in fig. (3-a,b,c,d,e,f.)

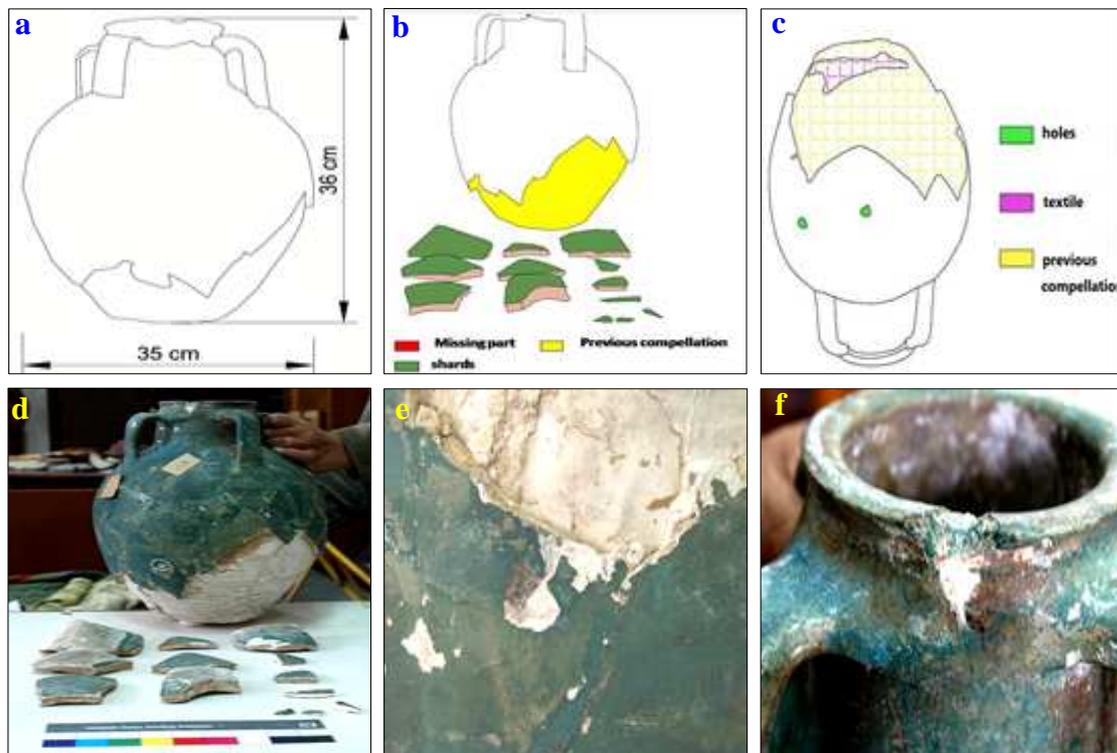


Figure (3) Shows the deterioration phenomena of the jar.

2. Analytical Techniques

The samples of the jar were examined by the naked eye as well as

using magnifying glasses $\times 10$ to identify the components in the fabric. In

addition, the same glasses were used to identify the components in the fabric of the core and the glaze. The mineralogical composition of the salts, the body, and the glaze layer was determined by XRD using x-ray powder diffractometer (Xí PORT PRO. PANALYTICAL) with Ni-filtered Cu K• target with secondary monochromator at operating conditions of 45 kV/40 MA. Furthermore, the firing temperature of the pottery body was estimated from the mineral phases obtained by X-ray diffraction. The SEM-EDX was used to determine micro-textural and micro-

chemical features of the body and the glaze layer. It was performed with JEOL 5410 Scanning electron microscope equipped with an Oxford (England) EDX Microanalysis system (25 KV, 0.28 no, ~1 µm beam diameter, 60s; counting time). Elemental analysis was obtained using the Oxford INCA-Energy software. Microstructure and chemical constitution of the sample was carried out by Scanning electron microscopy (SEM) JEOL, JXA- 840A, Electron Probe Micro Analyzer, coupled with energy dispersive X-ray spectroscopy (EDS) INCA- Sight.

3. Results

3.1. Visual examination

The sample was examined by the naked eye and by using magnifying glasses 10 × to identify the component of the fabric as an attempt to discover the structures, porous fabric and gaps. They were handmade not using the

wheel fine texture and reddish color. The glaze layer and the body were homogenous with subtle variations in the percentage of line intensities in the body. In addition, no slip layer could be observed.

3.2. XRD analysis study

The XRD analysis of crystallized salts and the body, fig. (4-a, b) indicated the presence of many minerals. The crystallized salts were Halite (NaCl) and Calcite. It was indicated that this clay contained Silica (SiO₂), Hematite (α-Fe₂O₃) Calcite and Gehlenite (Ca₂Al₂SiO₇). Gehlenite is calcium silicate originating from the thermal transformation of calcareous clays in an oxidizing atmosphere at a temperature ranging from 700-1050 °C. At higher temperatures,

it was transformed to another one that was not found in our sample [15]. The presence of gehlenite in addition to the diffuse presence of hematite might suggest the oxidizing atmosphere during ceramic annealing. The quartz in the ceramics could have a double origin; it could have been added as a temper, or it could have been formed during the annealing from the decomposition of the clay silicates [16].

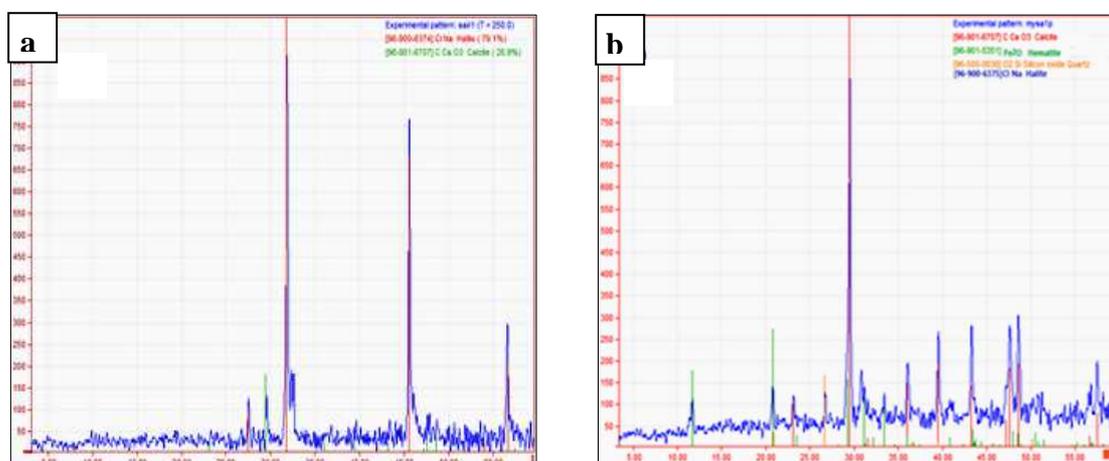


Figure (4) Shows XRD pattern shows **a**, the crystallized salts, **b**, ceramic body.

3.3. SEM-EDX study

By using the Scanning Electron Microscope, the internal structuring and homogeneity of the fabric could be determined. The body was ferruginous [16] with hematite content ($\alpha\text{-Fe}_2\text{O}_3$) (5.90 %). It was responsible for the red color of the body. In terms of calcium content, the body was made of highly calcareous clay; Ca (16.08 %), Silica (27.06 %), alumina (8.68 %), Alkali Na_2O (0.68 %), K_2O (1.16 %), and MgO (2.34 %). It contained coarse quartz grains surrounded by smaller sub-angular grains and very fine quartz grains in the glassy matrix. In addition, it had some gaps. Within the same context, quartz was abundant in the enamel base (2-32 %) and the alkali was mainly revealed in Barium BrO (8.89 %) which was greater than sodium (4.32 %) and potassium (2.06 %), (which probably proceeded from wood ignition). They formed the main alkaline raw material [17]. The jar was characterized by the absence of a

separately defined slip layer as the glaze layer was applied directly over the ceramic body. It could be manufactured in the same workshop due to similarities in materials and techniques, such as; the absence of slip layer, the deliberate addition of zinc oxide (ZnO) to the glaze mixtures and the higher amount of lime present in the glazes. Tin oxide (SnO) was identified in the glazes of jar in quite high amounts (4.77 %). The addition of zinc oxide to the glazes to improve their chemical resistance increased their reflective index and hardness. It could, also, be used to obtain shine and matte glaze [17]. Zinc oxide could be added to the glaze as Zincite (ZnO). In addition, Chromite (Cr_2O_3) was used in quite high amount (4.69 %) in the jar to obtain green glazes as it was used in Persian tiles to obtain green glazes [18]. EDX results are listed in tab. (1) and SEM features shown in fig. (5-a, b, c, d, e, f).

Table (1) chemical composition of ceramic body and glaze layer by EDX analysis.

Sample	Analytical Results												
	SiO_2	Al_2O_3	Na_2O	K_2O	MgO	Fe_2O_3	CaO	CO	Cr_2O_3	SO	Cl_2O	ZnO	BaO
Body	27.06	8.68	0.68	2.16	2.34	5.90	16.08	-	-	-	-	-	-
Glaze	2.32	3.22	4.32	2.06	0.34	1.25	11.36	12.98	4.69	13.24	1.41	4.77	8.89

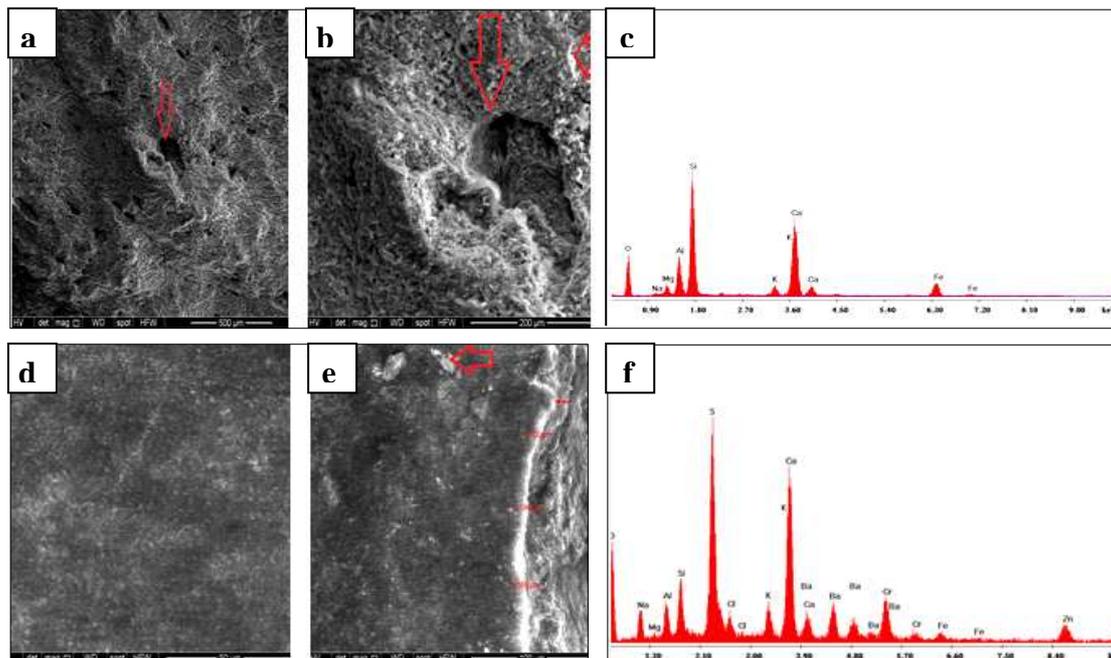


Figure (5) Shows investigation results **a.**, **b.** SEM photomicrographs, **c.** EDX spectrum of the ceramic body layer of the jar. , **d.**, **e.** SEM photomicrographs, **f.** EDX spectrum of the glaze layer of the jar

4. Treatment Processes

4.1. Cleaning process

Before performing the cleaning process, it was essential to identify the type of pottery, its mineralogical composition and the nature of dirt and deposits. It was also important to understand that cleaning was meant to remove soil or deposits and encrustation rather than the removal of original material or any opaque weathering crust or a patina. It had a protective action and archaeological feature [19]. The old restoration materials on the jar were required to pick or cut of the surface with 4.1.1. Wet cleaning.

In this case, mechanical cleaning techniques was not enough to remove the previous layer (incompatible restoration process); therefore a wet cleaning method was applied. Cotton poultices immersed in water were effectively used to soften

4.1.2. Removing soluble salts

In this case, the soluble salt was halite (sodium chloride). Mechanical cleaning tools such as brushes, needles and scalpels were used to remove crystallized salt and then cotton poultices immersed in water were used to extract the salt through the pores of object [13]. Ethylene di amine tetra-acetic acid (EDTA) was used to remove calcareous deposits. It was the best for removing calcareous material. According to many specialists [19,20,21] 5 % solution of the tetra-sodium salts of EDTA, that gave a pH of 11.5, was used to remove the thin layer of insoluble salts from the surface

the tool such as a needle, sharp scalpel, and wooden tools. The objects were carefully supported on a padded surface, only the minimum necessary pressure was applied and sharp tools could scratch the body surface; they were avoided. Removing insoluble calcareous deposits on the outer surface of the jar was partially carried out using abrading method. Glass fiber brush and dental brushes were successfully used. Because this object was relatively sound, satisfactory results were obtained

the hard crusts on the surfaces and made them easy to remove Then, different tools such as a needle, sharp scalpel were required to pick or cut carefully the hard deposits and old restoration [13].

without seriously affecting the iron content of the object. Different treatment processes of jar, cleaning of hard deposits, bonding of the broken shards located in the neck, the body and the base of the jar are shown in fig. (6). For example, during the burial period, the higher level of the ground water could carry the dissolved salts into the pottery pores, leaving them (dissolved salt) behind when the saline water evaporates [14]. The soluble salts are hygroscopic and as the relative humidity rises and falls, the salts repeatedly dissolve and crystallize [15].



Figure (6) Shows the jar during the restoration processes.

4.2. Bonding process

The broken shards located in the body of the jar were bonded by using Paraloid B-82 dissolved in acetone with concentration of 40 % as an adhesive. The adhesive was applied to one-half of the break only, and the two halves were brought together. Paraloid B-82 was suitable for bonding earthenware objects

4.3. Replacement process

Many parts of the jar were lost; including the base, rim and body. Unfortunately, the base was completely lost. Choosing the material that would be used to fill the missing area depended on the type of the ceramic object being treated and the method used to apply the filling material [13]. The missing parts were filled by using polyfilla (calcium sulfate with cellulose fibers). The filling materials could be applied to small chips and cracks without using any form of support. Larger fillings often required some forms of support for the filling material until they had been cured. Because the missing parts in the jar were large, to pottery burned powder (Grog) was used after the re-crushed to become softer until it became appropriate to the fabric of the jar. Homogenous granules were

4.4. Coloring

Because the missing parts in the jar were large, some coloring experiments were made with pieces of the dried dough to reach the appropriate color using acrylic reversible colors. The

and the drying time would depend on the ambient temperature, and the thickness and porosity of the body [13]. Different narrow and wide cracks on the jar surface were reinforced by using Paraloid B-82 adhesive where it was applied along the surface of the crack from one side with a fine paintbrush.

used as a support material through curing the filling material that was being used (polyfilla) [14]. Before applying the filler, the break edges were dampened first with water to avoid the rapid absorption of water from the filler [20]. Alternatively, the edges were sealed using Paraloid B-82 10 % to make the filling easier to reverse. Regarding the missing part of the base, the present part was used as a guide to form the new one. Grog (iron clay) was used and the similar missing part was formed by the freehand. It was dried in the open air. Finally, it was polished and fixed in its original place with Paraloid B-82 adhesive, as. A permanent consolidation treatment was carried out with 5 % Paraloid B-82 in acetone, which was applied by using the brush [22].

color complementary part of the artifact, and diluted water emulsion had used a mix of green acrylic color as could be seen in fig. (7).



Figure (7) Shows the jar after finishing the restoration processes.

5. Discussion

An Egyptian ceramic jar that dates back to the Mamluk period (1250-1517 A. C.) was discovered at Al-Fustāt; the first Islamic capital of Egypt. XRD analysis is carried out on the crystallized salts, i.e. Halite (NaCl) and Calcite. XRD analysis of the ceramic body suggests the presence of Silica (SiO₂), hematite (α -Fe₂O₃), Calcite and gehlenite (Ca₂Al₂SiO₇) that is a calcium silicate originating from the thermal transformation of calcareous clays in an oxidizing atmosphere at a high temperature of 700– 1050 °C. Tin oxide (SnO) is, also, identified. In general, quartz is the most common mineral phase in the raw materials. Its high content indicates that the used clay has a high proportion of silica that owes to the fillers or the composition of minerals or to adding quartz to clay as additive for its improvement [8]. Moreover, a proportion of red iron oxide Hematite (Fe₂O₃) is found; it works as a strong aid during the melting process. It, also, gives red color to the body where the final color of pottery depends on the chemical state of iron [9]. Via SEM-EDX, it can be noted that the samples are characterized by the presence of internal structuring composition and defined fabric. The body contains fine quartz grains in the glassy matrix, in addition to the homogeneity with the appearance of some small gaps, and the shape of grains in terms of coherence and the

absence of a separately defined slip layer as the glaze layer is directly applied to the ceramic body. [16] It is noted that the appropriate burning temperature of the ceramic body ranges from 700– 1050 °C, where the clay would essentially be an amorphous material with some residual grains of quartz [15]. It may be claimed that the properties of clay differ from one type to another according to the difference in chemical composition. Also, it is significantly affected by water [14]. Other features, such as the absence of slip layer, the deliberate addition of zinc oxide (ZnO) to the glaze mixtures and the higher amount of lime in the glazes suggest that the jar may be manufactured in the same workshop due to similarities in materials and techniques. Zinc oxide is added to the glazes to improve their chemical resistance and increase their reflective index and hardness. It can also be used to obtain shine and matte glaze [16]. It can be added to the glaze as Zincite (ZnO). Quartz is abundant in the enamel base and the alkali is mainly revealed in Barium which is greater than sodium and potassium (which probably proceed from wood ignition). They form the main alkaline raw material [17]. Although Green glazes are made by mixing blue and yellow mineral oxides, in our study Chromite (Cr₂O₃) is used in quite high amount (4.69 %) to obtain green glazes.

6. Conclusion

The jar was discovered at Al-Fustāt archaeological site during the season of 1945. It dates back to the Mamluk period (1250–1517 A.C.). Its shape and the degree of color were common in this period. It was obvious that this jar was subjected to an incompatible restoration process in the past. The result obtained by SEM/EDS for body sample showed a good agreement with the XRD result. The results obtained by different analytical techniques (XRD, SEM/EDS) showed that the clay used for producing the jar was rich in iron oxides 5.90%, Calcite and gehlenite (Ca₂Al₂SiO₇). The firing temperature of the jar was over 700 °C. Which was estimated from phase to phases determined by XRD analysis? In addition, Tin oxide (ZnO) was identified in the glazes of jar in quite high amounts (4.77 %). The addition of zinc oxide to the glazes improved their chemical resistance and increased their refractive index and hardness. It could also be used to obtain shine and matte glaze, Chromite (Cr₂O₃) was used in quite high amount (4.69 %) in the jar to obtain green glazes. The jar could have been manufactured in the same workshop due to similarities in materials and techniques, such as: the absence of slip layer, the deliberate addition of zinc oxide to the glaze mixtures

and the higher amount of lime in the glazes CaO (11.36 %). The jar suffered from many deteriorations, including incompatible restoration process and materials. In addition to cracks, flaking and lamination of body and re-crystallization of soluble salts within the pores were found. While mechanical cleaning tools such as brushes, needles and scalpels were firstly used to remove the old restoration and crystallized salt, chemical cleaning was carried by different solvents and EDTA solution. Mechanical cleaning tools were used to remove crystallized salt (halite) and then cotton poultices immersed in water was used to extract the salt. The broken shards were bonded using Paraloid B-82 dissolved in acetone with concentration 40 %. The missing parts were manufactured using Grog (powder of burned pottery) and Polyfilla (calcium sulfate with cellulose fibers) was used as a filler to replace the missing parts of the jar and it was fixed by Paraloid B-82 adhesive. After the restoration processes were completed, acrylic reversible mix green colors were used.

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