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Deterioration Aspects of the Egyptian Faience Ushabti Statuette of the King Aspelta kept in Atfih Magazine, Egypt

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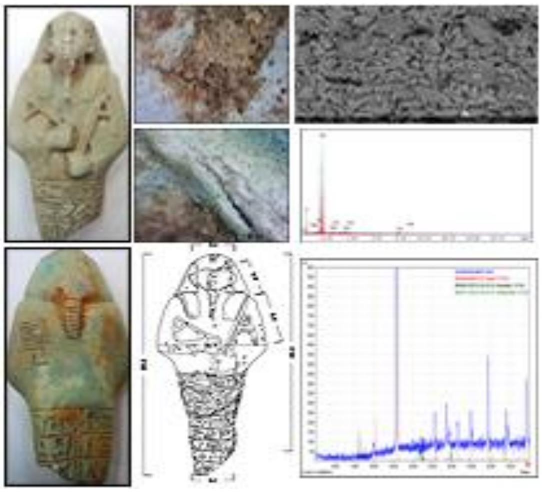
HIGHLIGHTS

- The Ushabti statuette of King Aspelta from Atfih, Egypt has great archaeological value due to its material and unusual size.
- The identification of the components of the statuette revealed information on raw materials, glazing method, and color materials.
- The study is mainly concerned with the characterization of the deterioration aspects of the faience statuette.
- The deterioration aspects found are color change, weakness, breaking, glazing detachment and accumulation of dirt and dust.

GRAPHICAL ABSTRACT



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ABSTRACT

The Aspelta faience Ushabti statuette is one of a group of statuettes kept in Atfih magazine, located 80 kilometers south of Cairo, Egypt. This store contains different types of artifacts excavated from different periods and sites in Egypt. This study aims at characterizing the materials and manufacturing techniques used to produce this statuette and its deterioration aspects. The analytical techniques used were AutoCAD, USB light digital microscope, X-ray diffraction (XRD), and Scanning electron microscope coupled with energy dispersive X-ray (SEM-EDX).

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The results revealed that the efflorescence method was used in the manufacturing of the glaze layer colored with Egyptian blue. Many degradation aspects such as discoloration, increase of porosity, cracks, surficial flaking, and salt crystallization have been observed. Moreover, the X-ray diffraction analysis proved the presence of the basic copper chloride compounds "Atacamite and Paratacamite", which led to the color change of the glaze layer, which may be due to the poor storage and improper preservation. It can be added that these results will help the conservators to produce some solutions for the conservation of these artifacts in the near future.

1. Introduction

The Aspelta faience Ushabti statuette was chosen from a group of statuettes that were originally stored in the Egyptian Museum in Cairo, and then transported to the Atfih magazine when it was built around 2004-2005 AD (Fig. 1).The Egyptian faience material may be defined incorrectly as ceramics or glass by its appearance, but in fact it has a distinctive composition of ground quartz or sandy body coated with a soda-rich glaze layer. It was first produced in both Egypt and the Near East as early as the 4th millennium BC and continued in production until the Roman period. The faience material was used to produce various objects such as Ushabti statuettes, bowls, tiles, and amulets, etc. Initially the principal colorants used were copper and manganese producing turquoise and black glazes, respectively. Then, with the beginning of glass production around 1500 BC, the glaze colors were extended to include cobalt blue, manganese purple and lead antimonate yellow [1]. It has been called the ‘first high-tech ceramic [2].The Egyptian faience consists of 92–99% silica (SiO_2), 1–5% lime (CaO), and 0–5% alkali, such as soda (Na_2O). Moreover, it contains smaller amounts of aluminum oxide (Al_2O_3), iron oxide (Fe_2O_3), magnesium oxide (MgO), copper oxide (CuO), and potassium oxide (K_2O), with trace amounts of other elements [3].Around 5 - 25% clay is added to facilitate the formation of the body [4]. There are three known methods of faience production: Efflorescence, Cementation, and Application [5].Efflorescence is the most popular method and was known as a self-glazing method [4].In this method, the alkali salts were mixed with particles of quartz and pigments. After the evaporation of the water the alkali migrates from the core to the surface with the color granules. A salty crystalline network and a thin layer of glaze is formed when the object was fired [6]. Faience was generally fired at temperatures of 800–1000^{O}C [3].

However, because of poor preservation and storage, all the faience statuettes kept at Atfih magazine were damaged and deteriorated. Furthermore, the fragments were stored together, which negatively affected the state of their preservation. Consequently, the Ushabti statuettes suffered from many deterioration aspects such as cracking, crystallization of salts, missing areas, and the color change of the glaze layer [7].One of the most important features of deterioration is the friability of the faience body. The strength of the core depends on the amount of glass present in the body. Once the glaze surface is lost the quartz in the body will be brittle, so cracking and fracturing will take place. Therefore, faience is always susceptible to more damage from physical shock, particularly if the glaze is thin or incomplete or deteriorated [8]. Another deterioration feature is the crystallization of salts. The condition of faience objects is at risk because they contain soluble salts or/and subjected to climate changes. All soluble salts will deliquesce at a certain level of relative humidity, depending on the type of salt, and recrystallize during drier periods. This process causes damage as the newly formed crystals occupy a greater volume than the salt solution and exert pressure on the pore walls. Faience material can be vulnerable to crystallizing salts, as they disrupt the glaze surface, or cause detachment of the glaze layer from the core by delamination [9]. It is a major reason for the color change phenomena of faience [10].Color alteration is a common phenomenon in ancient Egyptian blue faience. The presence of chlorides catalyzes the decay process and causes what is comparable to bronze disease [11].Moreover, it is also widely known as “copper chloride cancer". The deterioration of faience can be attributed to weaknesses in the original composition [8]. Dust and dirt accumulated on the glaze surface led to the blurring of the decorations and distortion of the surface of the faience object. (Fig. 2).The aim of this study is to identify the chemical composition and manufacturing technology of the Egyptian faience statuette of king Aspelta and its glazing technique. Moreover, degradation products were characterized to develop a future restoration plan for this statuette.

Fig. 1. Shows the unsuitable storage conditions of many Ushabti statuettes before the study

2. Archaeological background and description of the King Aspelta Ushabti.

Egyptian faience Ushabti (funerary figure) is one of the museum collections stored and preserved in the Atfih magazine. These statuettes were manufactured by the ancient Egyptians as grave goods. Ushabtis are usually mummy form in shape and originally represented the deceased in his dignified statuettes. The purpose of these statuettes was to serve the deceased in the afterlife. Whenever the deceased was summoned to cultivate the fields in the hereafter, a shabti was supposed to present itself on his/ her behalf saying, "I shall do it, here I am" [12]. They first appeared in the Middle Kingdom, when only one or two of them were placed in the tomb to do various chores on behalf of the deceased, towards the end of the New Kingdom, the number of shabtis per burial increased considerably [13].It is worthy to mention that statuette can be dated to the reign of the king Aspelta since it bears his royal cartouche [14] (Fig. 3). That king ruled the Kingdom of Kush from 593 -568 B.C, his time was concurrent with the reign of the Egyptian King Psammetik II, the fourth king of 26thDynasty [15].On the other hand, the statuette was stored in a wooden box together with a group of broken statuettes which led us to suppose that these broken statuettes were associated with the campaign that was despatched by the Egyptian King Pasmetik II to Napata the Capital of the Kingdom of Kush according to two fragmentary stelae from Karnak and Tanis. Furthermore, the Egyptian army destroyed the city of Napata [16]. Additionally, the destruction of Napata led the king Aspelta to move the capital to Meroe [17]. Since it continued as a capital till the end of kush Period in the 3rdcentury B.C [18]. This royal Ushabti is of light blue glazed faience. It depicts a mummiform figure wearing a royal nemes headdress and a long plaited false beard. Both hands rest on the chest, right above left, holding implements of field work. The right hand holds a small hoe, and the left holds a hoe and a cord to a bag slung over the shoulder. Eight horizontal lines of hieroglyphic text appear on the legs, with traces suggesting they were filled with black color. The text records a version of the "Shawabty Spell" for the king [19]. The nose and uraeus-cobra on the headdress are both chipped. The lower portion of the legs is broken and fragmented. Some small fragments are missing. The dimensions of the studied statuette, the length is more than 26 cm, and the width is more than 9 cm.

3. Materials and Methods

Five samples were used in the investigation of the faience statuettes. Very small fragments were taken from the body and the glaze layer of the statuette from areas that do and do not display color change. These samples will help in identifying the components and deterioration products of King Aspelta statuette.

The following methods and techniques were used to collect the data, through which the aims of the study were achieved:

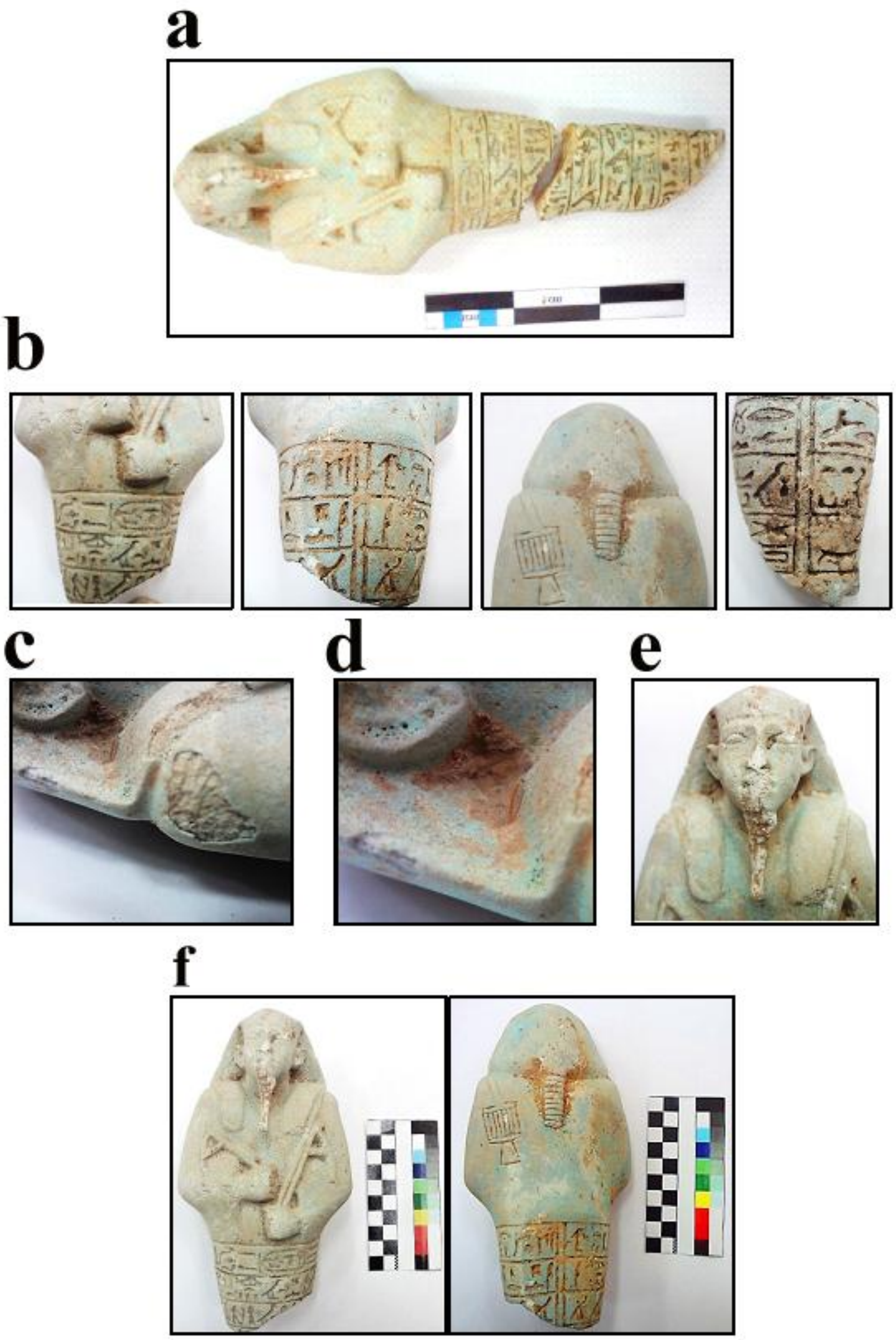

Fig. 2. Shows deterioration aspects of the King Aspelta faience statuette, a.General view with broken area at the middle and loss of the pedestal, b.Dust and dirt on the surface and inside the hieroglyphic text, c. Loss in the glaze layer, d. Pits in the glaze, e. Loss in the noise and false beard, f. Fading the blue color and salt crystallization.

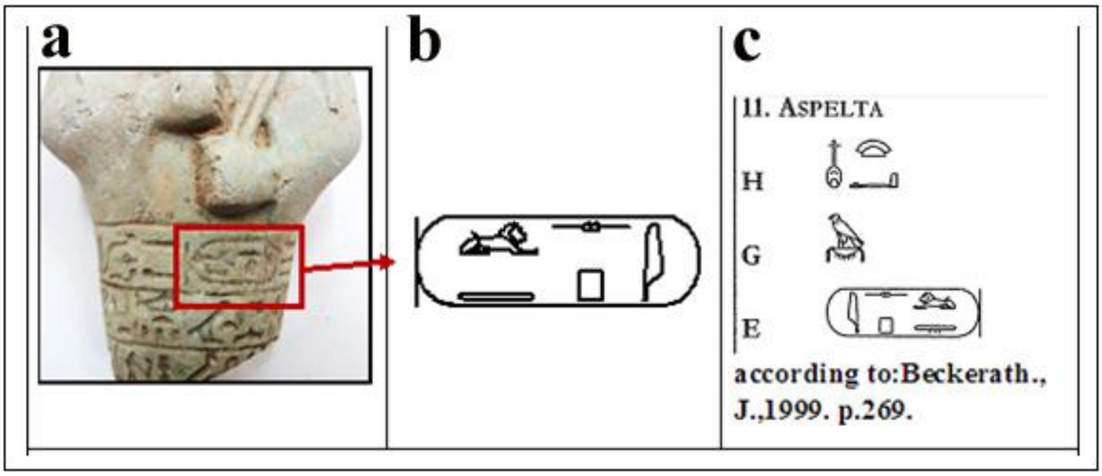

Fig. 3. Shows the royal cartouche and identification of the name of the statuette's owner, a. The royal cartoucheon the front side, b. Transcription the cartouche, c. Searching references to know its owner.

3.1. AutoCAD documentation

AutoCAD program version 2020 Autodesk company was used to document the dimensions and visible deterioration aspects [20].

3.2. USB Digital Microscope

A portable optical digital microscope (Image sensor 1.3 Megapixels, magnification factor 10~1000 times, photo capture resolution 640×480, 320×240, and LED illumination light re-source adjustable by the control wheel) was used. It is non-destructive and easy to apply. It presents a means of studying deterioration features and damage phenomena that are not clearly visible with the naked eye such as micro cracks, voids etc. [21].

3.3. X-Ray diffraction

X-Ray diffraction analysis (XRD) was used to identify the mineralogical composition of the body, the color of glaze and the color change of the Egyptian faience [22]. XRD device with the following operation conditions was used: *Diffractometer type: PW1840, Tube anode: Cu, Generator tension (KV): 40, Generator Current (mA): 25, Wavelength Alpha1 (Å): 1.54056, Wavelength Alpha2 (Å): 1.54439, Intensity ratio (Alpha2/Alpha1): 0.500, Receiving slit: 0.2, Monochromator used: NO*.2θ scanned range (5 - 60°).

3.4. Scanning Electron Microscopy (SEM)attached with EDX

A scanning electron microscope attached with Energy Dispersive X-ray spectroscopy unit (Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V., using 14-x up to 100000 magnification and resolution for Gun. was used to identify the body and glaze layer's physical properties, type, colors, and chemical composition through the attached analysis unit [23] [24].An area analysis was carried out in the analyzed samples.

4. Results and discussion

4.1. AuoCAD documentation

Auto-CAD was used to document the dimensions,(Fig. 4 a) and the deterioration features of the statuette, (Fig. 4 b). The maximum length of the statuette is 26.2 cm, and the maximum width is 9.7 cm.

4.2. USB Digital Microscope

The examination by the portable optical light digital microscope showed different deterioration aspects of the Ushabti statuette(Fig. 5). It revealed the accumulation of dust and dirt, as well as cracks, loss in the glaze layer and voids that are widely spread on the surface of the statuette. Flaking and the presence of pores in some parts of the glaze layer were also detected. The backside of the statuette displayed black stains, color alteration, and the formation of salts crystals on the surface(Fig. 6).

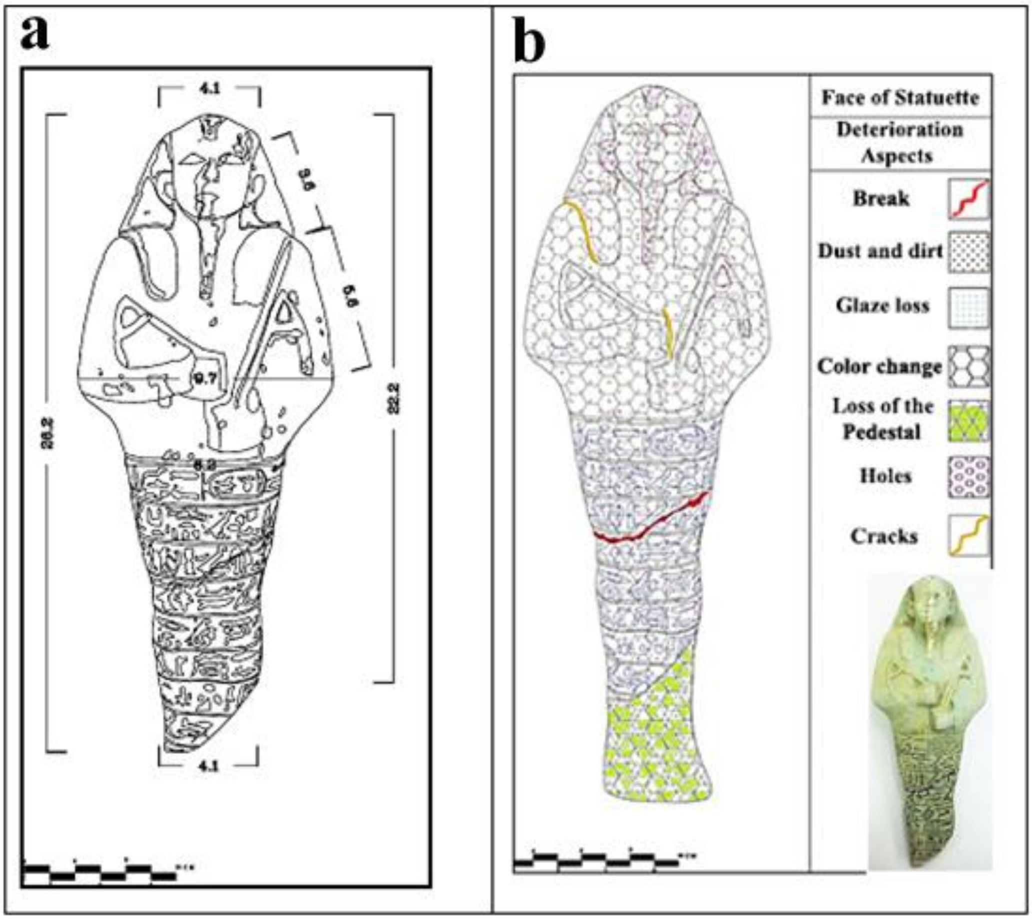

Fig. 4. Shows geometric documentation of the King Aspalta statuette, a. The dimensions, b. Deterioration aspects of the statuette

The USB digital microscope showed some defects in the glaze, such as pinholes due to the improper manufacturing of the glaze layer. The most common cause of pinholes in Egyptian faience is the presence of gases that form on the glaze surface in the form of bubbles. During firing, gases escape from the body and produce bubbles, penetrating the surface. At the beginning of glaze melting, bubbles develop from the gases released by the decomposition of organic compounds and carbonates (CO_2, CO), sulphur dioxide (SO_2), or from air and moisture trapped inside the glaze[25]. These bubbles burst and create holes inside the glaze, known as pinholes. These holes are scattered in many areas on the surface of the statuette and reduce the glaze layer efficiency. The USB microscope also showed the buildup of dirt and dust on the surface and inside the hieroglyph letters, resulting in the deformation of the glaze layer, due to the burial in the soil.

Moreover, a lot of cracks and loss of the glaze layer were detected. Cracks in the glaze can lead to the loss of the layer's properties and may facilitate the penetration of liquids, resulting in severe damage of the faience[26]. Additionally, crystallized salts were detected on the backside of the statuette. Faience contains a percentage of the "natron salts" in its composition that was used as a flux [27]. These natron salts contain more than 30% sodium chloride, which is a soluble salt [28] and the method of glazing by efflorescence depends on these salts [29]. Porous faience can also deteriorate due to the presence of soluble salts within the body itself where the salts dissolve and re-crystallize. The salt crystallization results in great pressures within the pores of the body or at the glazed body interface area where this causes the glazed layer to flake away from the faience surface [7]. On the other hand, is a major source of the manifestation of the color change phenomenon in the faience at high humidity [9].

4.3. *X-Ray diffraction*

XRD results of the samples showed that the core of faience statuette consists of quartz (SiO_2) only as a major phase, (Fig. 7 a). While the XRD results of the glaze layer sample showed that it consists of quartz (SiO_2) as a major mineral phase, Cuprorivaite ($CaCuSi_4O_{10}$) and Calcite ($CaCO_3$), as minor components, (Fig. 7b). Finally, the diffractogram of the glaze layer that displayed discoloration showed that the sample consists of quartz (SiO_2) as a major phase, and atacamite and paratacamite ($Cu_2Cl(OH)_3$) basic copper chlorides as minor phases (Fig. 7c).

The results of the X-ray diffraction of the faience statuette core showed that, the sample consists mainly of quartz SiO_2, a known component of the composition of Egyptian faience body. Generally, the body consists of silica up to 99% [30]. The results of the analysis of the second sample of the glaze color indicated the presence of about 67 % Quartz SiO_2, 23.3% Cuprorivaite" Egyptian blue" $CaCuSi_4O_{10}$ and 9.4% Calcite $CaCO_3$.This indicates the use of Egyptian blue to obtain the blue color of the glaze layer. Cuprorivaite $CaCuSi_4O_{10}$ was used as a blue pigment for the faience tiles in the south tomb of King Djoser in Saqqara [11]. Egyptian blue is a multicomponent synthetic blue pigment that has been recorded in ancient Egypt [31]. It is an artificial pigment made by heating together silica, copper alloy filings or a copper ore such as malachite, lime, and an alkaline material such as natron or potash "flux" [32].

The XRD result from the sample of the glaze layer that displayed color change, showed the presence of 74.7 % Quartz SiO_2 as a major component, 13.7 % Atacamite and 12.3 % Paratacamite $Cu_2Cl(OH)_3$ basic copper chlorides. This caused a change in the blue color of the statuette [10].The color alteration is one of the most identifiable of ancient faience damage [22]. It is known as 'Copper chloride cancer that provide a degradation process of Egyptian faience in conjunction with copper-based pigments [33, 34].

4.4. Scanning Electron Microscopy attached with EDX

SEM-EDX analysis has been used by many researchers to study Egyptian faience, as it provides information on the chemical composition as well as the material's morphology[3][5][6]. The examination of the sample from the faience statuette core by SEM showed the heterogeneous components; Quartz grains appeared in different shapes and sizes with large intergranular spaces between the grains. The EDX results of the core sample revealed that Si is the main element, whereas Mg, Al, Cl, Ca, Fe, and Cu are formed low percentages (Fig.8 a)and Table 1. The SEM of the glaze layer showed that the micro-cracks are spread on the surface in addition to the presence of pores, pits, and losses in the glassy phase as well. The EDX results of the same sample demonstrated the presence of different percentages of Si, Na, Mg, Al, S, Cl, Ca, Fe and Cu (Fig.8b and Table 1).

SEM-EDX analysis of the glaze layer provided essential results on the composition of the faience statuette, glaze type and color oxide. Table 1 shows the presence of Si, Al, Na and Cl elements in the core and glaze layer. This indicates that the faience consists mainly of quartz and alkali. The Si and Al indicates the presence of quartz and aluminum silicates- the base of the glaze [35]. Also, it indicates the use of natron as a flux material "alkaline glaze" due to the presence of about (1.36% Na) and (1.88 % Cl) in the glaze layer [28]. For the inner core, it shows the presence of copper (3.66%Cu), this indicated that the faience was produced by the method of self-glazing "Efflorescence" [36].In this method the materials of the faience body (paste) are mixed with the coloring material (frequently copper). After firing in the kiln, this effloresced coating forms the colored glaze layer. Due to the mixing of the components throughout the body materi- to fuse together and create a glaze layer on the surface [2].

SEM-EDX analysis of the glaze layer, detected the presence of copper (10.69% Cu), with a percentage of silica (38.69% Si), calcium (2.04% Ca) and (2.36% Al). The presence of these elements in the glaze layer indicates that the blue color of the statuette resulted from the use of "Egyptian blue. On the other hand, the presence of chlorine (1.36% Cl) with copper ion (Cu) in the glaze layer, caused the main color change and the high humid atmosphere as well [11].The process begins with the devitrification of copper and chlorine bearing glass and glaze. Then, al, rather than simply adding them to the surface of an already made object, the firing causes them copper and chlorine ions are leached out of the residual glass and gel into migrating solutions. Basic copper chloride (atacamite or paratacamite) precipitates through reactions between the leached copper ions with sodium chloride-rich solutions. Basic copper chloride deposits on the surface of the glaze layer, mainly in its gaps. The original blue color of the paint or glaze changes therefore to faint green. This process leads to the development of highly friable and spongy pigment layers or glaze thus resulting in their disintegration to fine powder, denudation, and ultimately final destruction [33].These outcomes confirm the results of the X-ray diffraction.

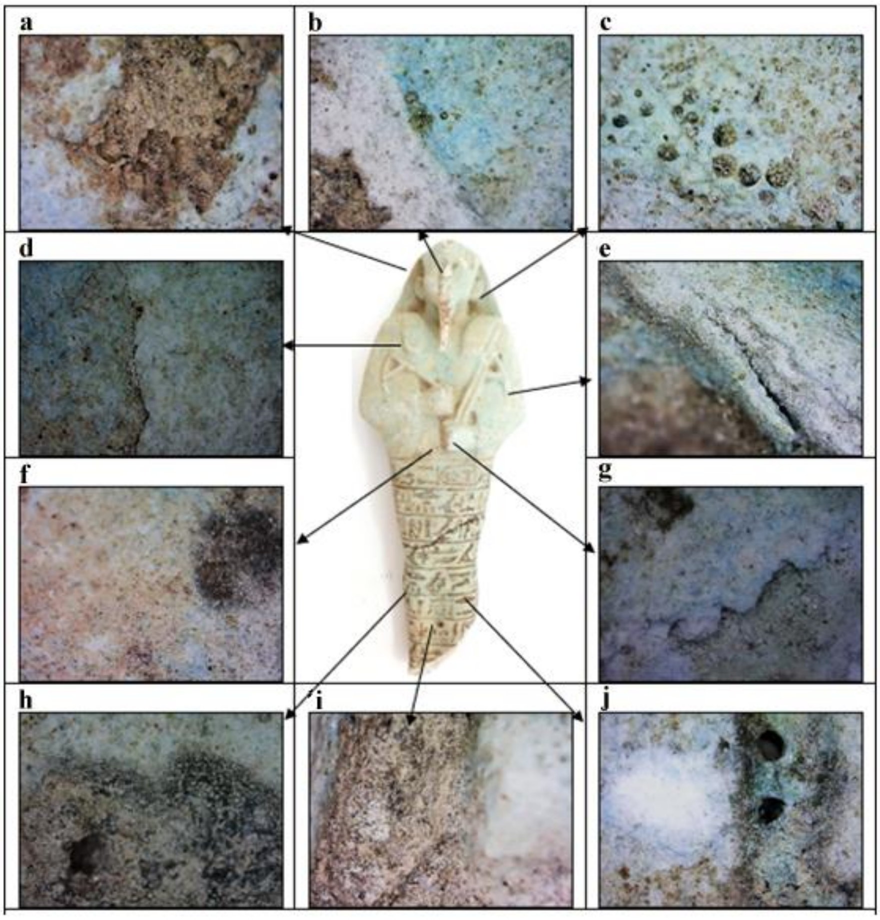

Fig. 5. Shows the examination by USB digital light microscope (the front), a. Dirt and dust penetrating the surface, b. Loss of glaze, c. holes in the glaze layer (manufacturing defects), d. Micro-cracks, e.Glaze flaking, f. Black stains in the glaze, g. Detaching and peeling of glaze layer, h and i.Dust and sand inside the hieroglyph's letters, j. Big holes with dirt

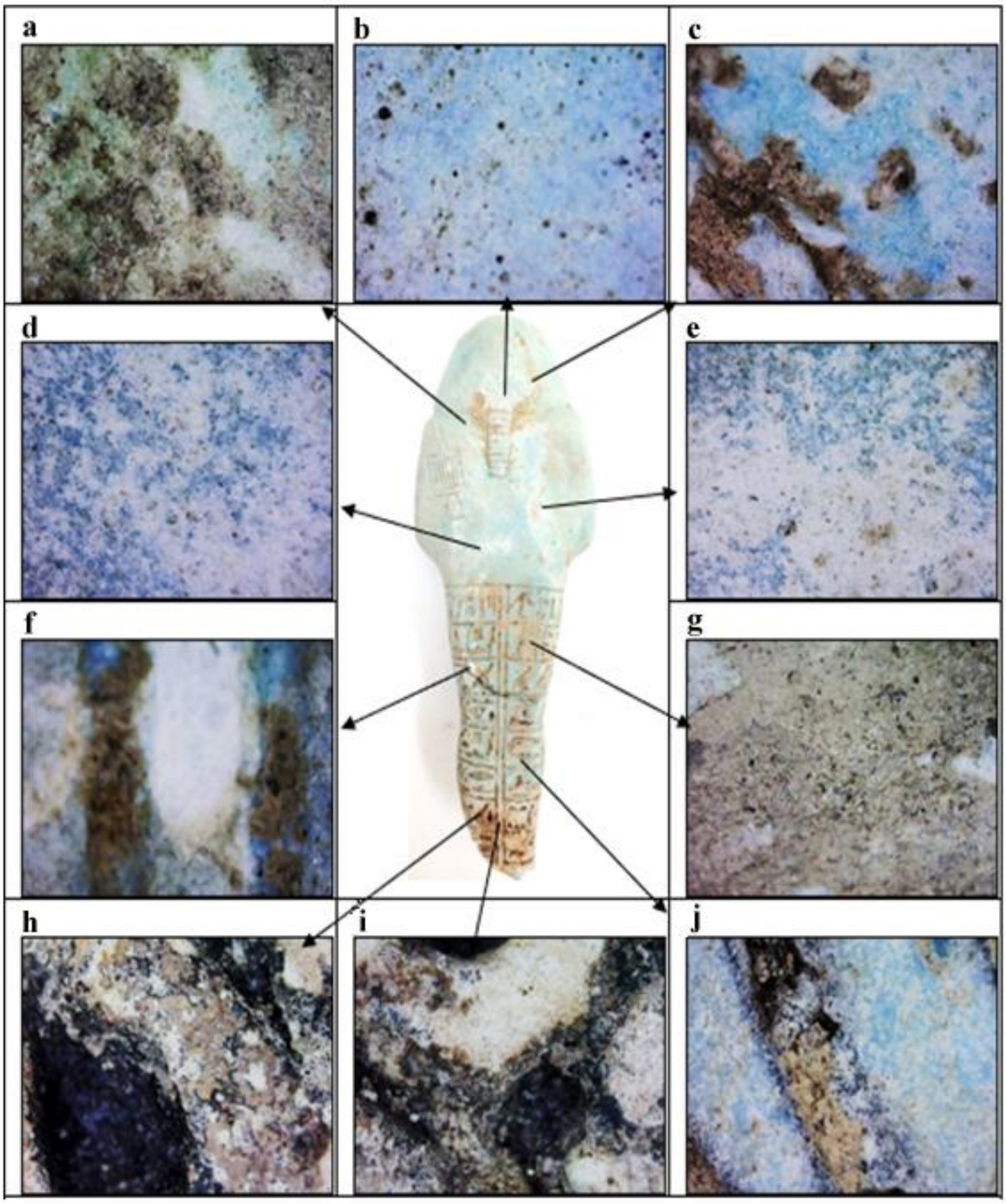

Fig. 6. The examination by USB digital light microscope (The backside of the statuette), a. Dirt and color alteration, b. Holes in the glaze layer (manufacturing defects), c. Dust and sand inside the glaze, d and e. Salts crystals on the surface, f. Glaze flaking, g. Dirt and dust sticking to the surface, h and i. Brown dirt layer firmly adhered to the glazing surface, j. Remains of the black color inside the hieroglyph's letters.

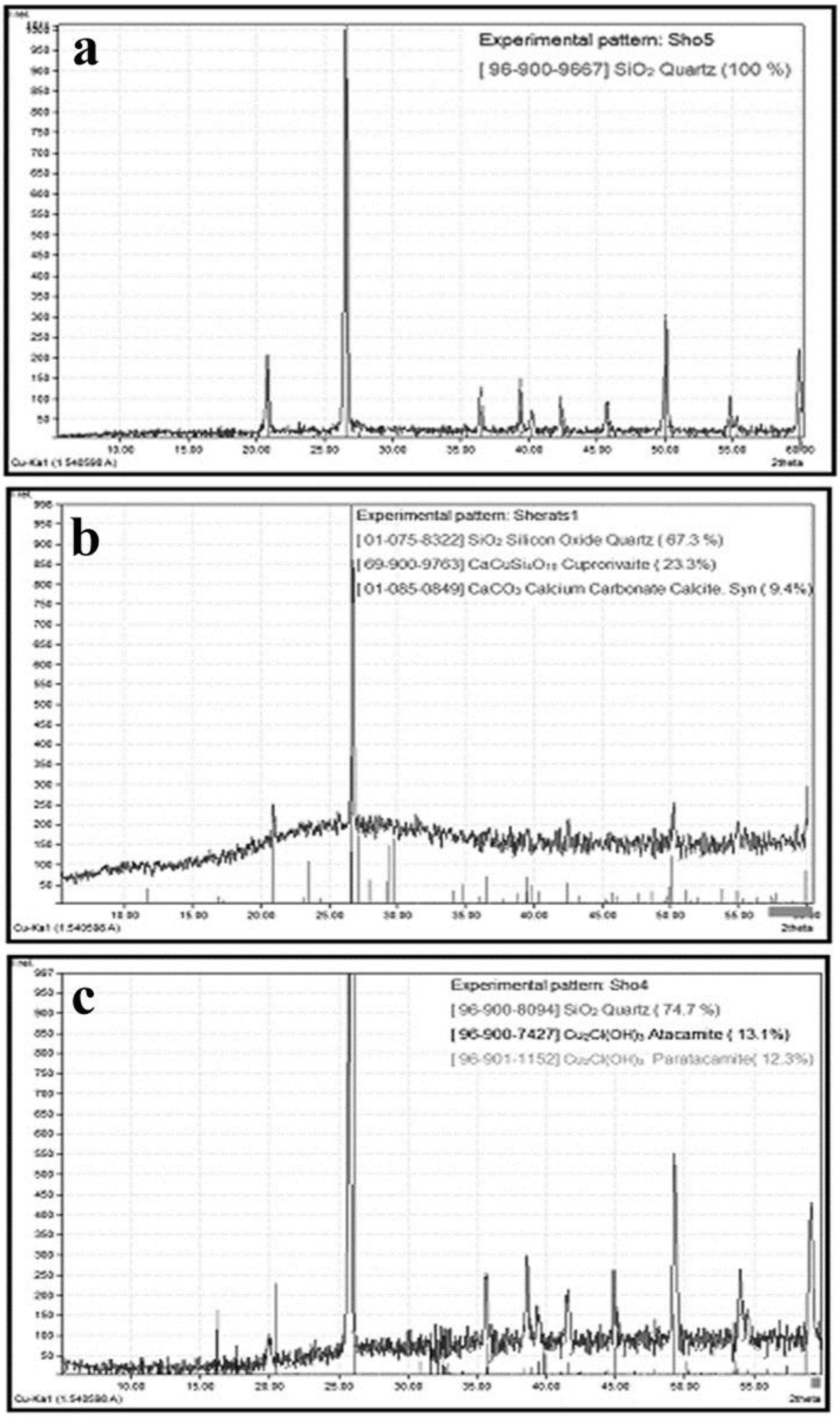

Fig. 7. XRD patterns of faience samples, a. Pattern of the core, b. Pattern of the glaze color, c. Pattern of the glaze discoloration

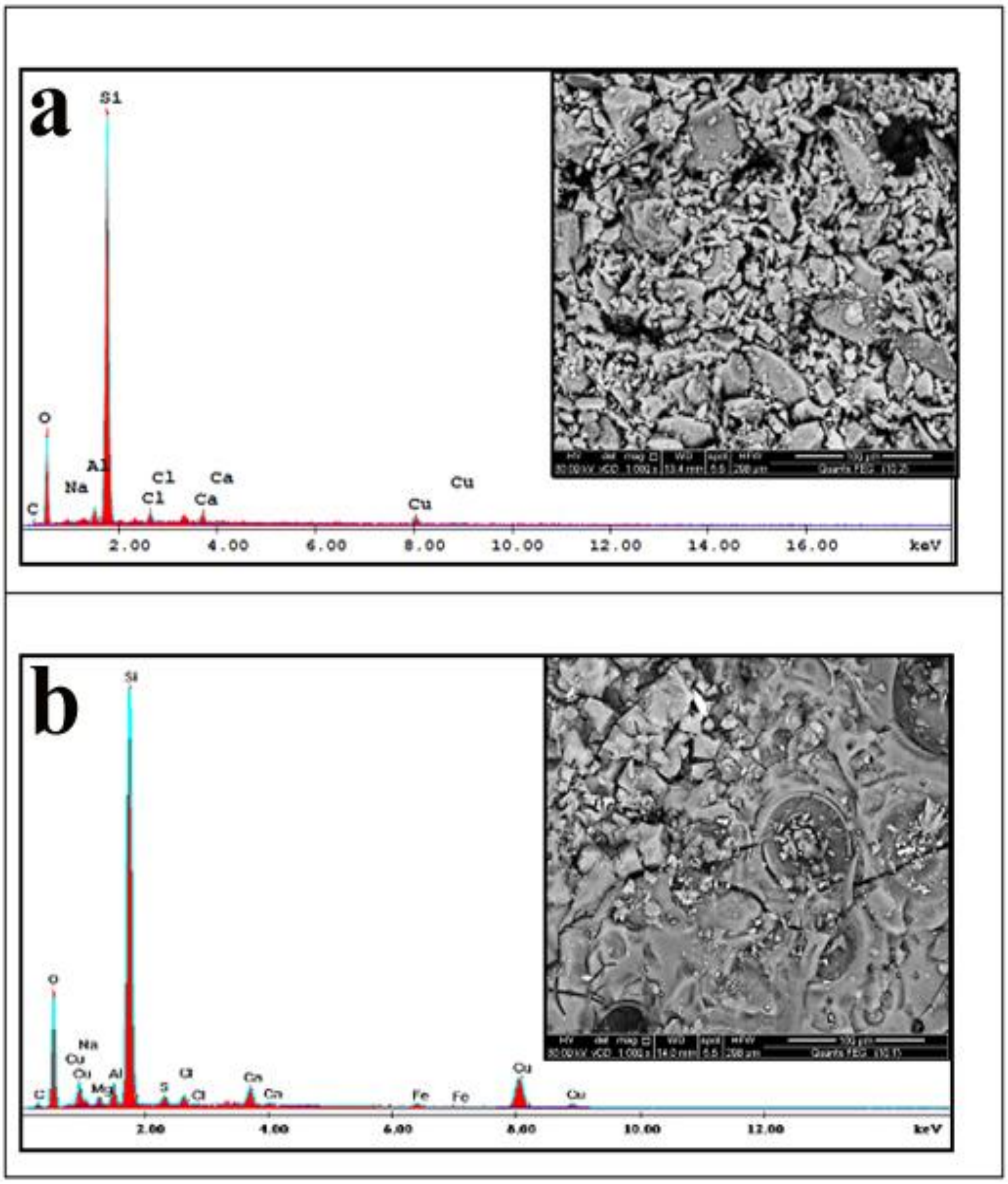

Fig. 8. Shows SEM/EDX results of the faience, a. The core, b. The glaze layer

Table 1. EDX wt% values of the elements present in the core and glaze layer of the faience statuette

Weight percentage of the elements in the samples %												
Sample	**C**	**O**	**Mg**	**Na**	**Al**	**Si**	**S**	**Cl**	**Ca**	**Fe**	**Cu**	**Total**
The core	**7.71**	**37.01**	**….**	**1.66**	**2.02**	**44.56**	**----**	**1.88**	**1.51**	**….**	**3.66**	**100%**
Glaze layer	**6.84**	**33.64**	**1.25**	**1.36**	**2.36**	**38.69**	**1.18**	**1.14**	**2.04**	**0.80**	**10.69**	**100%**

5. Conclusion

This research investigated the damage manifested by the Egyptian faience Ushabti statuette for the king Aspelta and the characterization of its state. It is made of blue faience. It is unusual to find an Ushabti statuette that measures 26.2 cm in length and 9.7 cm width. Therefore, this Ushabti is unique and has a historic and aesthetic value. Hence, it represents one of kings that ruled the Kingdom of Kush. On the other hand, it has a special dimension in comparison with the other faience statuettes. Therefore, it was necessary to study one of the rare sizes of these objects. This study displayed important information about the components and manufacturing techniques of the statuette. Efflorescence (it is known as the self-glazing method) was the method used to produce the glaze layer. The Egyptian blue was used as a pigment to obtain the blue color of the statuette. However, due to manufacturing defects, various pinholes were detected in the glaze layer, which weakened this protective layer. In addition, inadequate storage, high humidity, and poor preservation in the Atfih magazine increased the deterioration process. The cracks, holes and color alteration have diminished the protective qualities of the glaze layer, increasing its weakness and degradation. It is well known that the glaze layer is the protective layer that gives the products strength and stability. Here the glaze layer is damaged, so the structure and binding force of the Ushabti is at risk, as it was weak and broken. The present study throws light on the relationship between the production technique and degradation process of the Egyptian faience. Hence, it offers the museum curators and restorers many benefits in this field to understand this relationship. Especially, when making decisions about storing, handling, and treating faience objects. The study will be useful for the protection and museum display of faience objects in Egypt.

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