ORIGINAL PAPER

THE INTERACTION BETWEEN A WHITE MATERIAL INSIDE AND OUTSIDE A POTTERY VESSEL FROM EASTERN EIN-SHAMS, EGYPT

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Abstract. *This work aims to investigate a pottery vessel from eastern EIN-Shams, Egypt, lying in sandy soil from citizen lands, returning to the New Kingdom-Egypt. This pottery vessel was covered with white material from inside and outside. In terms of origin, this vessel is made of buff-colored clay; it has big pores, many cracks, and large missing areas filled with that material, with an uneven surface texture, that strongly bonds with the body pores, and shrinks away from the edges in some areas, in this way obscuring the original surface. In this respect, several physicochemical investigations in terms of chemical composition, physical properties, and nature of this white material were performed, to analyze the influence of it on the ancient pottery vessel. The analytical methods used in this research were USB digital microscopy, X-ray diffraction (XRD), scanning electron microscopy coupled with energy dispersive X-ray (SEM-EDX), and differential thermal analysis (DTA). The results of the USB digital microscopy showed the presence of gypsum crystals, large, flaked pottery surfaces, and quartz. SEM-EDX revealed the presence of organic fibers, chloride salts in a significant percentage, iron, and aluminum oxides. XRD investigation emphasizes the presence of gypsum and sodium chloride salts. DTA results revealed that the firing temperatures of the pottery vessel range between around 750-800°C.*

Keywords: *Pottery vessel; microscopy; XRD; SEM-EDX; DTA.*

1. INTRODUCTION

The problem of this study is that a pottery vessel was covered and filled with white material - as found in its burial in citizen lands in eastern EIN-Shams (Egypt). It is among the most famous archaeological sites in Egypt; it is considered an important excavation area in the northeast of Cairo. Ein-Shams is one of the oldest neighborhoods in Cairo and forms a part of the ancient city of Heliopolis/ancient Egyptian Ounu [1]. In addition, it contains many historical excavated areas, like the New Kingdom (about 1550-1069 BC), and the third intermediate period for the Ramesside (20^{th}-24^{th} Dynasties) [2] (Fig. 1).

Painting on pottery was uncommon before the New Kingdom but often appeared in it, especially in the late 18^{th} Dynasty. Vases can be ornamented with colorful floral designs; cobalt blue is well-known and distinctive in this kind of product [3]. According to the 19th - 20th dynasties, the vessels could be uncoated or unpainted but sometimes had red, cream/pink, and white slipped, probably comprising the most familiar pottery of the New

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Kingdom [1]. The surfaces of the pots were treated in many ways; one of them was that they were easily smooth while the clay was still wet [2].

This type of jar is distinguished by its rounded, slightly internally thickened rim [1], many jars with rounded or pointed bases (as in most Egyptian pots) have an interior ledge below the rim [2]. In addition, the studied vessel's body is made of buff-colored clay, containing organic temper, made of Nile clay fabrics: almost Nile B2 [1], which is for the rougher variant vessels [4]. Nile clays are more common than Marl clays [5]. The vessel was wheel-turned- fabrics of wheel-turned pottery appear in the examination of the break that is parallel to the rim [6], handless, and covered with thin creamy slip. The most characteristic types among the Nile clay forms of the 18th Dynasty are round based, which can be associated with the tradition of this period [5]. In addition, the bases of most Egyptian pots are round [2]. The vessel has many cracks, big pores, and some missing areas filled with that unknown white material. This material has an uneven surface texture, with strong bonds with the pores of the body. It has shrunk away from the edges in some areas, especially in the larger areas. It has been smeared over the edges, obscuring the original surface around the fills. This vessel was lying in sandy soil; the main reason for that was the change in the burial customs. Understanding pottery's characteristics and how they can deteriorate, as well as following basic safety precautions, are required for providing pottery objects with the correct maintenance [8], or the reconstruction of pottery distribution [9].

Figure 1. EIN-Shams archaeological site on Egypt map.

2. MATERIALS AND METHODS

2.1. MATERIALS

The presented samples of the pottery body and the white material covering it were chosen from an excavated vessel returned to the New Kingdom (about 1550-1069 BC) and prepared to a 0.3 mm grid before applying the analyses. Understanding the properties of ceramic artifacts can help care for them correctly [8] (Figs 2a and 2b). DTA gives a complete database based on classification by color, materials characterization, and evaluation of their deterioration [10].

(a) (b)

Figure 2. Shows (a) The studied pottery vessel. (b) The white material inside the pottery body vessel.

2.1. METHODS

It was essential to identify the nature, chemical composition, and micro-structural characteristics of this filling & coating white material, which smeared the initial and outer surface of the chosen pottery vessel, to know the reaction extent between it and the vessel. This extent depends on its industrial development and the firing degree of the vessel. When an artifact is being treated, micro-excavation and examination are included in the definition of conservation [9, 33]. Mineralogical and chemical investigations revealed the pottery object's raw material [7]. Recognizing all this information facilitates the choice of safe conservation techniques and suitable materials for removing and cleaning dirt and other restoration steps [11], and to study a representative sample's mineralogical and chemical composition from the body pottery vessel. Moreover, important technological information is provided by the mineralogical composition that relates to the manufacturing process and firing. Furthermore, the characterization of artifacts such as shape dimensions, binders' aggregate ratio, and additive components affect the physical properties of the body vessel [11, 12].

Different microstructural analytical techniques, such as USB Digital Microscopy, X-ray Diffraction Analysis (XRD), Differential Thermal Analysis (DTA), and Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDX) are used.

2.1.1. USB Digital Microscopy

The USB Digital Microscopy is a non-destructive technique, that appears in the external physical state [13], using an adjustable microscope working station (super eyes) at x10 to x500 magnifications. This investigation worked out at the Department of Conservation, Faculty of Archaeology, Cairo University, Egypt.

2.1.2. X-Ray Diffraction Analysis

This analytical technique is performed by using a Philips analytical X-ray system (Ni-filter, tube anode Cu, monochromatic used No). X-ray Diffraction Analysis was used to investigate the chemical composition and the crystalline components of the pottery vessel and the white material samples, in addition to the color and damaged phases that may exist in the chemical composition [7]. This analysis was performed at the General Authority for Mineral Resources in Dokki, Egypt.

2.1.3. Scanning Electron Microscopy

SEM-EDX is a non-invasive technique used to investigate the morphology of samples [14] as well as to examine the proportions of minerals that differed in the fired samples [15]. This investigation was performed by using a Model Quanta 250 FEG (Field Emission Gun) microscope coupled with an EDX unit (with accelerating voltage 30 K.V., magnification 14x up to 1000000x, and resolution for Gun.1n). This investigation was achieved at the Microanalysis Center from Cairo University, Egypt.

2.1.4. Differential Thermal Analysis

DTA provides important information regarding the crystalline structure of the target compound and the level of hydration of inorganic salts. Through DTA, there is a noticeable decrease in weight in the pottery samples [12]. The estimated firing temperatures of the pottery vessel are ascertained through the application of differential thermal analysis; the thermal transformation in the constituent mineral of clay is considered irreversible [16]. The basic properties of the DTA instrument used were Detector: "DTA-50", Acquisition Time: 10:38:27, Atmosphere: "Nitrogen", Cell: "Platinum", Flow Rate: 30.00 [ml/min]. This analysis was carried out at Egypt's General Authority for Mineral Resources in Dokki, Egypt.

3. RESULTS AND DISCUSSION

The fill and coat white material smeared and distributed the vessel surface, which penetrates inside the pores. Moreover, dust exists on the vessel surface from both sides and small cracks beneath the white material because of the previous faulty restoration. It is very important to use advanced technologies to identify the elemental composition of archaeological ceramics [17], specifically the chemical analyses that define the structural characteristics of the degradation agents, required to select a suitable restoration material and techniques [18].

3.1. USB DIGITAL MICROSCOPE

Samples investigation in reflected light conditions is considered to be a method of digital documentation. In addition, it shows the petrographic structural features and the deterioration forms [11, 12]. The vessel body matrix contains inorganic additives like big grains of quartz (silica) and grog (grinding fired pottery) powder, in addition to straw as an organic temper. The white material covers and fills the pottery vessel.

The USB digital microscope took the surface topography of the pottery vessel matrix shown in Figures (3a and b). It contains a high amount of quartz, appears in big crystals, and has an abundant percentage of organic materials; some of them burned during the firing process, leaving large pores, and other organic material still exists in the form of straw [9, 32].


(a) (b)

Figure 3 (a), (b) USB digital microscope images show the surface topography of the pottery vessel, and the inhomogeneous degradation on the sample surface – act in organic materials like straw, and large pores, big grains of white material, and quartz.

The images also show the interaction between the texture of the pottery vessel and the white material that is clearly in the crystal and flakes of white materials on the surface and in the clay matrix. Figures (3c and d) show the white material's surface topography, which covered and filled the pottery vessel with a USB digital microscope; it appears in big and clear flakes and crystals. Also appears the pottery vessel flakes that adhered with the white material.


(c) (d)

Figure 3 (c), (d) USB digital microscope images show the surface features of the white material sample, It's big clear flakes and crystals. In addition, the pottery vessel flakes in buff/colored clay. This confirms the interaction between it and the white material.

3.2. X-RAY DIFFRACTION ANALYSIS

X-ray diffraction analysis gives two patterns: one returns to the pottery vessel and the other returns to the white material; the following crystalline mineralogical composition is known from the provided information of this process [12]. X-ray diffraction analysis pattern (Fig. 4a): the presence of quartz (SiO_2) 54.5% - microcline ($KAlSi_3O_8$) 15.2% - hematite (Fe_2O_3) 7.1% - gypsum ($CaSO_4 \cdot 2H_2O$) 22.9%.

From the result, it appears that the presence of microcline ($KAlSi_3O_8$) is the fundamental component of clay mineral consisting of the pottery vessel, hematite (Fe_2O_3) as a natural color oxide mineral to the clay mineral, in addition to a high percentage of quartz as an additive to the clay mixture [13]; that means high porosity to the pottery vessel. The white material in this sample is gypsum in a high percentage that interprets the deep penetrating of gypsum (the white material) in the pores of the pottery vessels cover and fill.

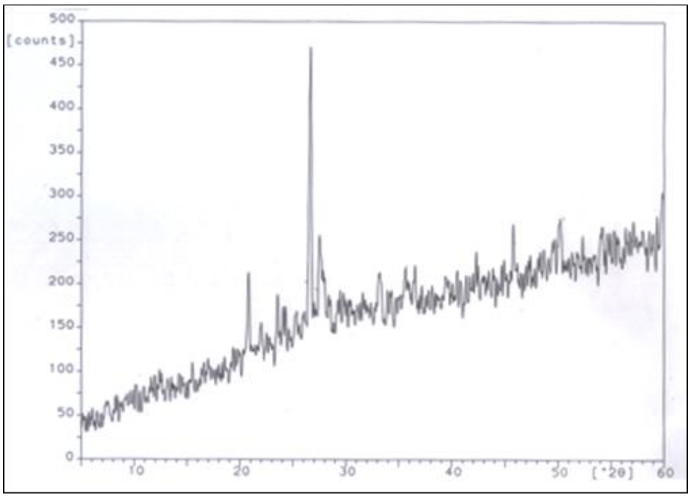


Figure 4a. Shows X-ray diffraction analysis pattern to the sample of the pottery vessel crystalline components.

X-ray diffraction analysis pattern (Fig. 4b): the presence of quartz (SiO_2) 33.1% - gypsum ($CaSO_42H_2O$) 28.8% - halite (NaCl) 17.7% - hematite (Fe_2O_3) 12.5% - magnetite (Fe_3O_4) 7.6%. SEM-EDX and X-ray can analyze the chemical structure of thick inorganic incrustation deposited during the long term [19, 7].

From the result, it appears that the white material that covers and fills the pottery vessels is gypsum with a high percentage of quartz (sand)- originating from very fine sandy inclusions [7], in addition to a high percent of halite (sodium chloride salt), which has a harmful effect on the body vessel by dissolving and crystalline [13]. A proportion of the hematite mineral was transferred from the pottery body to the gypsum deposits on it.

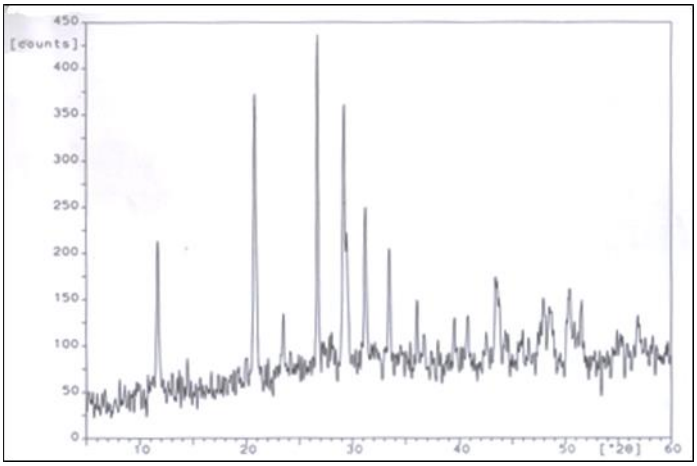
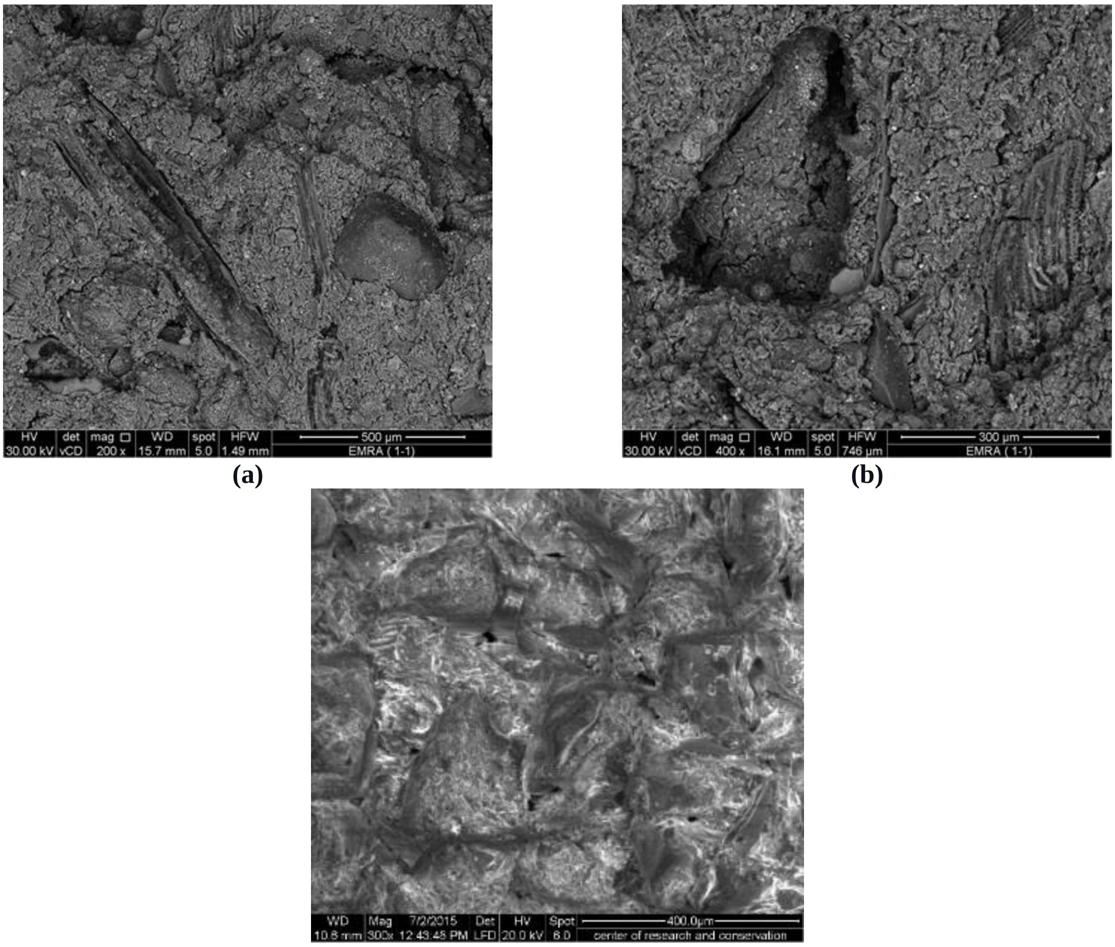


Figure 4b. Shows X-ray diffraction analysis pattern to the sample of the fills and covers material on the pottery vessel, which appears it be gypsum (white material).

3.3. SCANNING ELECTRON MICROSCOPY – ENERGY DISPERSIVE X-RAY SPECTROSCOPY

The details of pottery vessel surface morphological and micro-structural characteristics were detected by SEM-EDX. It contains residue fibers of straw (organic additive material) after firing, crystalline compounds in the pottery matrix, and some crystals of halite. The micromorphological and micro-structural characteristics details of the pottery vessel surface were revealed by SEM-EDX [15], and give information for interpreting slips, washes, and other surfaces [6]. The gypsum crystal also appears, confirming the interaction between the cover and fill white material (gypsum) and the pottery vessel matrix on the surface and in the pores. That appears in Figures (5a, b, c). The interpretation of the mineral identification results was confirmed by the X-ray diffraction analysis [11, 12].



(a) (b) (c)

Figure 5. (a), (b) SEM images show the surface texture of the pottery vessel containing crystalline compounds phases and inhomogeneous degradation compounds X200, 400, (c) The intrusion of gypsum deposits into the depth of the pottery pores X300.

The result obtained by SEM-EDX analysis revealed the basic technologies related to obtaining pottery and ceramic objects, as well as the type of clay mineral components [20]. Fig. 6 and Table 1 present the constituent elements of the clay matrix.

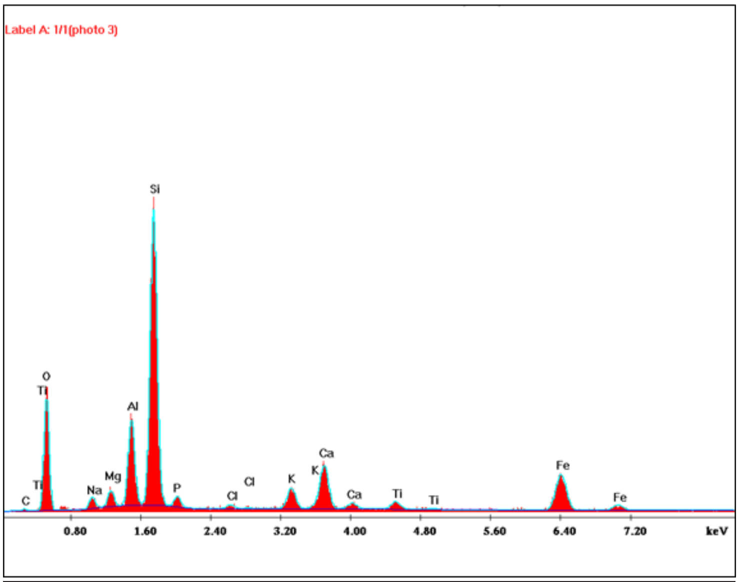


Figure 6. EDX spectra of the pottery vessel sample show the mineral elements that exist in the clay matrix.

Table 1. The concentration of elements [wt. %] by EDX to the pottery vessel sample.

Element	**C**	**O**	**Na**	**Mg**	**Al**	**Si**	**K**	**Ca**	**Cl**	**P**	**Ti**	**Fe**	**Total**
wt. [%]	2.57	36.01	2.15	2.10	9.01	30.14	2.24	4.75	0.42	1.51	1.06	8.04	100

3.4. DIFFERENTIAL THERMAL ANALYSIS

It is essential to understand the thermal changes (drying, dehydration, decomposition, combination, inversion, and vitrification) before working out the firing curve of DTA [23]. A significant weight of the clay body is lost at the first drying stage of the absorbed water [22], called the green body (unfired) and it is first dried. However, it may contain around 1 to 2.5 percent moisture before entering the kiln; this moisture is evaporated before the temperature reaches 200°C [23]. The moisture is driven out of the clay during the firing process [21]. At different temperatures, DTA curves show various endothermic and exothermic peaks. Moisture is responsible for the endothermic peak around 100 to 200°C [16]. Then, the organic substances contained in the clay matrix were carbonized and combusted at temperatures between 300 and 500°C. Thermal investigation reveals that endothermic peaks appear from 550 to 650°C [12]. The high weight loss between 50 and 650°C is because of the moisture decrease, which is identified through the endothermic peak in DTA [16].

The organic substance carbonized at 300 to 500°C is oxidized from about 800°C, this reaction has caused an exothermic peak of the analysis [22]. Firing starts partly at the end of the oxidation period; the strength increases slightly over that of the grey clay body [23]. All these actions interpreted the clay matrix shape of the vessel, in addition to the large pore size of burned organic materials, which add to the clay mix, and the high percentage of added silica. There is an endothermic peak at 500°C associated with the decomposition of clay minerals such as chlorite and montmorillonite [22]. The fired product's temperature is increased until it fully melts [23]. From the DTA chart, it appears that the firing temperatures of the pottery vessel range between around (750-800°C), which were defined as a well-fired vessel [24] (Fig. 7).

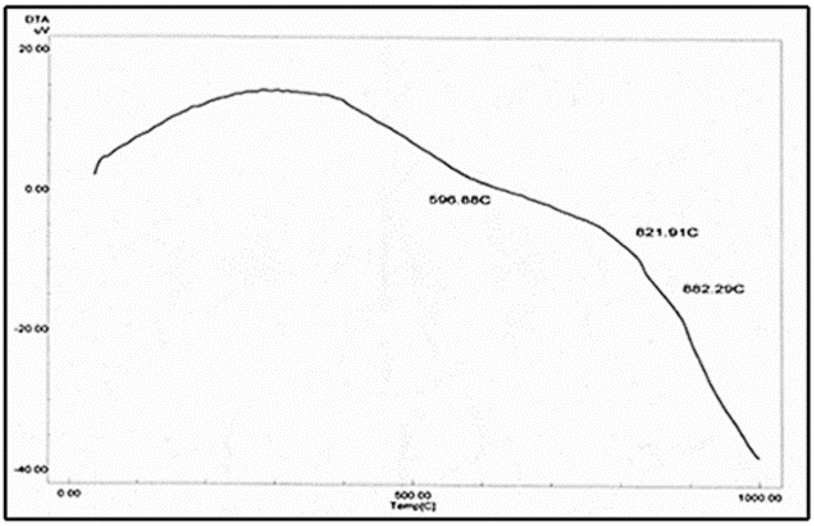


Figure 7. DTA Chart shows the heat curve of the studied pottery vessel sample.

3.5. DISCUSSION

The study presented significant results, related to the microchemical and microstructure of the vessel investigated by different methods such as USB digital microscopy, SEM-EDX, XRD, and DTA.

USB digital microscope shows the surface topography of the pottery vessel and the aspects of surface degradation that appear in big pores resulting from burning organic materials like straw; in addition to inorganic additives such as grog powder, large grains of quartz and gypsum as sediment in the pores (white material $CaSO_4 \cdot 2H_2O$). Clay is the essential ingredient in ceramic compositions, which provides plasticity. One of the methods for controlling firing shrinkage is the use of additives beyond which the strength decreases [25]. The use of the USB digital microscopy image shows in reflected light conditions the surface features of the white material sample, its big, clear crystals, and crusts, in addition to the pottery vessel flakes in brown color. This confirms the interaction between it and the white material.

XRD determined the mineral phases in the samples [26, 27] and identified any crystalline components that were present in the pottery body [28]. It appears that the white material, which covers and fills the vessel, is gypsum deposits, which reflects the deep penetration of it in the pores. The basic component of clay minerals is microcline, which consists of pottery vessels, and hematite is a natural color oxide mineral of the clay mineral. In addition to a high percentage of quartz as an inorganic additive to the clay mixture [24], that means a high porosity of the pottery vessel. Moreover, a high percentage of halite has a harmful effect on the body vessels by dissolving and crystalline. The percentage of hematite was transported from the pottery body vessel to the gypsum. The gypsum, in the case of this vessel, may return to prepare whitewash in one of the workshops in that period, which was applied sometimes on pottery objects or other kinds of material. According to Arias K. (2021), thick layers of white substances, whether mortar, plaster, or gypsum, come from diverse contexts found in some pottery vessels [29].

The micro-morphological and micro-structural characteristics details of the pottery vessel surface were detected by SEM-EDX [30, 31]. The results revealed that it contains residue fibers of organic materials after firing like straw, crystalline components in the pottery matrix, and crystals of halite. In addition to the crystal of gypsum, which confirms the interaction between the cover and filling white material (gypsum) and the pottery vessel matrix on the surface and in the pores, these results were confirmed by the X-ray diffraction analysis interpretations. SEM-EDX helps determine the production techniques, and characterization of pottery objects [7]. The careful examinations of micrographs of white material reveal that the particles were in a heterogeneous overlap with many incisions [15]. Before removing any surface deposits, the chemical nature of it should be established [32].

From the DTA, it appears that the firing temperatures of the pottery vessel range between around (750-800°C), which means that it is a well-firing temperature since active sintering takes place during this period [23]. The vessel suffers from many deterioration features like weakness in the vessel body– it has a very high porosity – and is more sensitive to soluble salts.

Four perspectives are used to handle archaeological material: composition and processing technology; altering and degradation reasons and mechanisms; conservative intervention; case studies and examples of restoration and conservation [33]. Black gypsum crusts form especially on porous materials that can retain moisture within them [34, 35]; Particles from the atmosphere trapped in a gypsum matrix usually set up black crusts [36]. When utilizing metallic tools to remove gypsum deposits, extreme precautions must be exercised, so as not to scratch the object's original surface, keeping what is left of the patina that originally formed and covered it [37]. Deposits frequently obscure important decorations and details of pottery, thereby affecting its aesthetic, social, and historical values [18]. Wet poultices removed the remaining residue and traces of gypsum with tiny particles with a dilute solution. The chemical solution for a long exposure time softened the hard surface layers and penetrated the gypsum base in the bas-relief surface. The technology of laser cleaning is frequently used in the restoration and preservation of cultural artifacts [33].

Depending on the deposit being removed, the mechanical methods' cleaning effectiveness; basically, the different methods used to clean are divided into chemical, mechanical, and occasionally laser approaches. AB57 has been used to remove gypsum black crusts that have formed [34]. It is a mix of both ammonium bicarbonate $(NH_4)HCO_3$, and sodium bicarbonate $NaHCO_3$, used in low concentration 2-3%. The use of chelating agents like Calgon (sodium hexametaphosphate) has proven gentle and effective in removing concretions [36]. As for the different kinds of ceramics in Egypt, petrography, in conjunction with other different analyses, offers a means of gaining a better understanding of the origins of ceramics, along with general information on shaping methods and firing temperatures [6].

4. CONCLUSIONS

Pottery and ceramics offer great information regarding fabric classification, and petrographs should be included in the first phases of findings processing. Thick layers of gypsum (calcium sulfate) cover the archaeological pottery vessel of the study, which may be a vessel to prepare plaster sheets or white slips in the workshop areas. The white layers filled and in contact with body pores with the insoluble salts of calcium sulfate. USB digital microscopy shows the vessel texture and the big flakes and crystals of gypsum on and in it. According to SEM-EDX findings, the vessel body contains organic fibers spread in the matrix, and the filling material is interred in its pores. Gypsum is a white material in the form of fill and cover, containing impurities, clarifying the advantage of SEM-EDS analysis in provenance studies, particularly in the instances of course and diverse ceramic fabrics. The high percentage of gypsum in this sample emphasized by using XRD reflects the deep penetrating of gypsum in the pores of the pottery vessel. Halite (sodium chloride salts), in addition to iron oxides, and primary hematite, gives the vessel a red color. DTA appears the firing temperatures of the pottery vessel range between around (750-800°C), which means that it is a well-firing temperature. After mechanical and chemical removal of the gypsum deposit, the vessel should be stored or displayed in stable conditions, around 50 % relative humidity (RH), with no more than ±5 % variation in a day and suitable temperature. Free of pollution-which causes further damage.

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