ORIGINAL PAPER

STUDY AND CHARACTERIZATION OF OLD KINGDOM PERIOD POTSHERDS FROM ABUSIR EXCAVATION. A CASE STUDY

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Abstract. *The current work describes the different methods used to investigate various potsherds. The potsherds were discovered at the Abusir archaeological site, dating back to the Old Kingdom period. This research aims to study the chemical and mineralogical composition of the potsherds and salt crystallization. For this purpose, the analytical study was carried out using different techniques such as a digital and polarizing microscopy, scanning electron microscopy coupled with X-ray energy dispersion (SEM/EDX), and X-ray diffraction. Microscopic examination shows that potsherds suffer, from different deterioration aspects, such as salt crystallization, accumulation of dust, and black spots. The results of the polarized microscope proved that sand, grog powder, and calcite were used as additive materials to improve clay's properties. The EDX and XRD methods used in the current work provide an essential chemical view of the structure of the samples. The results of EDX confirmed the presence of a high percentage of chloride salts, iron, and aluminium oxides. XRD results revealed that the firing temperature of the potsherds is high, around 850-950 ^oC. Therefore, the hardness of these sherds is high. There is little doubt that this will aid in conserving this valuable pottery.*

Keywords: *potsherds; old kingdom; abusir; deterioration; polarized microscopy; SEM/EDX; XRD.*

1. INTRODUCTION

Pottery is a important subject for art-historical research because it always reflects current tastes. At different times, by different people, and for different purposes, different forms and embellishments were used. As a result, archaeologists can date archaeological sites by studying pottery [1-4]. Pottery has an essential and diversified function in archaeology, serving as a key to understanding many aspects of the evolution of human civilizations [3-5]. Additionally, pottery, the most frequented archaeological discovery, may be utilized to learn more about the site, the people buried there, and the major religious activities of their descendants who visited the region in later centuries [6-9]. Pottery is utilized to give a quantitative examination of materials that can help us comprehend Egypt's economy and civilization qualitatively [10]. Pottery is also frequently used for gathering and storing drinking water, grain storage, and cooking. This emphasizes the significance of pottery in both economic and social activity [11].

The potsherds were discovered during the excavations of Abusir in December 2014, within the mission of the Faculty of Archeology, Cairo University, Egypt. Abusir, one of the most important graves of the Old Kingdom Kings, is situated about 30 kilometres south of

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Cairo, on the western bank of the Nile [12]. Abusir is an important archaeological site from the Old Kingdom, located several kilometres north of Saqqara (29.54°N, 31.12°E) [13]. During the Old Kingdom's 5th Dynasty, the town of Abusir became the focal point of the famous western [14]. The colossal pyramids and other buildings of the 4th Dynasty had filled up Giza, forcing the 5th Dynasty pharaohs to explore elsewhere for their funerary monuments [15]. The Abusir location became a vital burial complex for royal court officials of the Third–Sixth Dynasties in the Third Millennium BC and lasted into the First Intermediate Period [16]. During the Old Kingdom Fifth Dynasty, Abusir became the focal point of the renowned western burial procedures operating out of the then-capital of Memphis, which stretched from north of Giza to below Saqqara [17]. The existence of Abusir Lake, which made the location conveniently accessible by boat, must have played a significant part in the selection of this site for the construction of ancient state tombs [18] (Fig. 1).

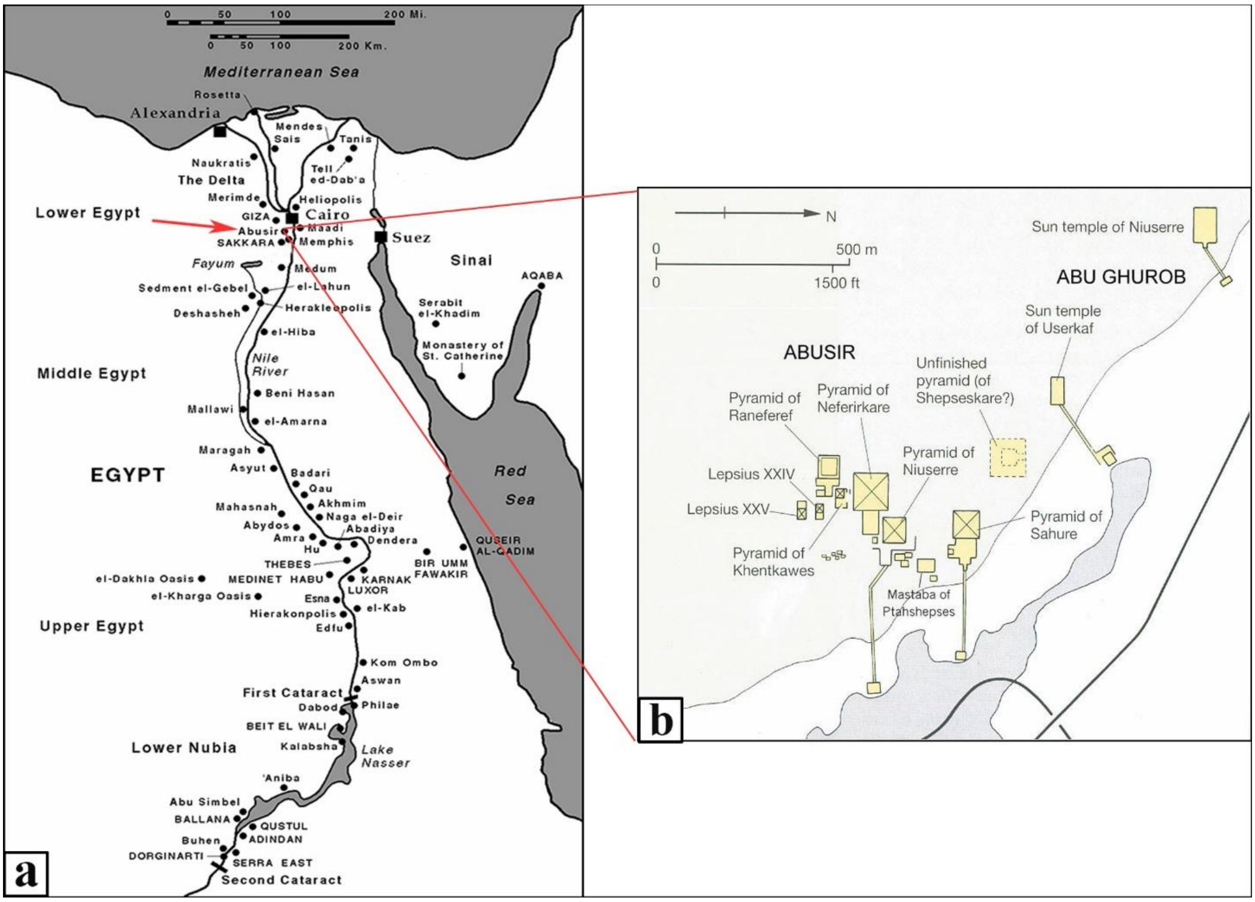

Figure 1. (a) Egypt map; (b) Abusir archaeological site.

The archaeological pottery buried in the soil is severely damaged due to soil compression, chemical deterioration, and the existence of salts [19-23]. Because archaeological pottery is formed of heterogeneous mineral components and has a low resistance to mechanical shocks, the most common deterioration aspects of pottery buried in the soil are breakage and surface distortion. This explains the large number of potsherds found in the archaeological sites [24]. The pores of the potsherds have an intensive impact on their physical and mechanical properties. These sherds have high water absorption ability, lower pressure-resistant, and a salt attack [25]. In addition, water can deteriorate pottery by causing cycles of freezing and thawing inside the pores or by causing the crystallization of salts inside the body transported by water from the environment [26, 27].

It has been known that inorganic porous materials such as pottery are susceptible to deterioration by salts. Therefore, the crystallization of salts depends on the size of the internal pores [28]. The salt could be a component of the soil or perhaps a natural mineral found in the pottery. The growth of salt crystals inside the pores of the pottery causes internal pressures on the outer surface, which may cause the slip layers to separate [29]. Analytical methods are essential for the conservation and restoration of artworks because they provide valuable information about such valuable artefacts' physical and chemical structures [1]. Examining and analyzing the pottery is essential in determining the causes of deterioration. It is also possible through various methods of examination and analysis to identify the pottery's mineral composition and determine the archaeological pottery's firing temperature [30].

In addition, these investigations enable us to determine the technological methods associated with pottery making [31]. These activities are nearly as effective in the research region through chemical and physical mechanisms. For example, groundwater and soluble salts are chemically major causes of pottery damage [32]. The potsherds suffered from many damaged aspects, including crystallization of salts on the surface, flaking of the slip layer, and the fragility of the pottery body. Besides, cracks of different sizes are scattered in the pottery body and the accumulation of dust and sand on those surfaces. Some parts of the slip layer were separated, and black spots were scattered on the surface. This paper aims to study the potsherds' chemical and mineralogical composition and the various damage forms that affect the sherds, either chemically or physically, caused by the effects of the surrounding environmental conditions. Finally, this study provides valuable information for preserving archaeological pottery extracted from excavations.

2. MATERIALS AND METHODS

2.1. MATERIALS

Three pottery samples of different shapes were chosen from the Abusir excavation and were used in the examination and analysis carried out in this investigation. These sherds are distinguished by their red color due to the use of a red wash layer. Red pots are the most common in that region [33]. Pottery vessels were also commonly used to store and preserve drinking water and cereals [8]. In addition, the thickness of the potsherds is about 0.5-1.0 cm

2.2. METHODS

Visual Assessment

The surfaces of the samples were observed using a SONY-type camera. These are the camera's specifications (16mm CMOS Sensor-8X Optical Zoom), Full HD movie (1920 * 1080) with up to 16x Clear Image Zoom and a 25mm wide-angle lens (35mm format).

Digital and Polarized Microscopy

Digital microscopy are performed using PZ01, USB 2.0 connector, Linux, Mac OS X 10.5.5 and above, at 10x to 500x. On the other hand, polarized microscopy was used to investigate the mineral appearance and define the sample firing temperature [24]. In this respect, ECLIPSE LV100POL (DS-FI1) polarized microscope (Nikon Instruments Inc, Melville, NY, USA) were used. Thin sections were made and examined using a polarized microscope in the micro analytical center at Cairo University's, Faculty of Science.

Scanning electron microscopy with EDX

The topography of the sherds was performed by scanning electron microscopy coupled with energy-dispersive X-ray analysis [3], using Quanta 250 FEG (Field Emission Gun) SEM coupled with EDX (Energy Dispersive X-ray) system, with a 30 K.V. accelerating voltage, magnification of 14x up to 1000000, and resolutions for Gun.1n); FEI is a Dutch corporation. This investigation was done at Egypt's General Authority for Mineral Resources in Dokki, Egypt.

X-ray diffraction

Mineralogical analysis acquired by X-ray diffraction (XRD) of the archaeological pottery can extend helpful information about raw materials and define the firing temperature, in addition to identifying deterioration aspects [34]. This investigation was performed using a PANalytical Multi-Purpose diffractometer, model X'Pert PRO (Analytik Ltd, Cambridge, UK), with secondary monochromator, Cu-radiation (1.542) at 45 kV, 35 mA, and a scanning speed of 0.04 °/sec. The relative intensities (I/I_o) and spacing (d) of diffraction peaks were measured between 2 and 60°. The relative intensities and diffraction charts are obtained and compared to the ICDD files. This analysis was performed at the General Authority for Mineral Resources in Dokki, Egypt.

3. RESULTS AND DISCUSSION

3.1. VISUAL ASSESSMENT

Visual assessment is considered the initial step in the examination procedure. It is recommended to employ a variety of lenses to evaluate the status of pottery, functional pottery usage, and the nature of the deterioration [25]. The visual examination of those potsherds reveals that the salts were spread on the outer surface. Moreover, black spots are scattered on the surface. In addition to the presence of dust and sand adhered to the surface of the potsherds resulting from the burial for an extended period in the soil. In addition to separating the slip layer from the surface, which is linked to the crystallization of soluble salts following rapid excavation at the archaeological site [31] (Fig. 2).

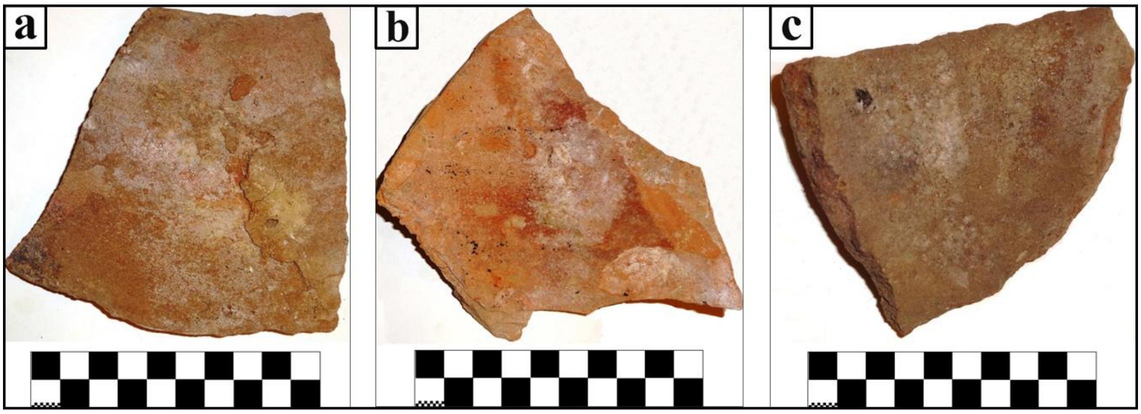

Figure 2. Shows the studied pottery sherds. (a) Sample (P1), (b) sample (P2), (c) sample (P3).

3.2. DIGITAL MICROSCOPY

The digital microscopy is considered one of the essential tools available to the restorer, and its images assist in evaluating the deterioration aspects of objects [35]. The digital microscope revealed various aspects of the potsherds' damage. This deterioration has represented the crystallization of salts on the outer surface and the accumulation of dust and sand on the surface. It also reveals the presence of pits on the body and black spots scattered on the surface (Fig. 3).

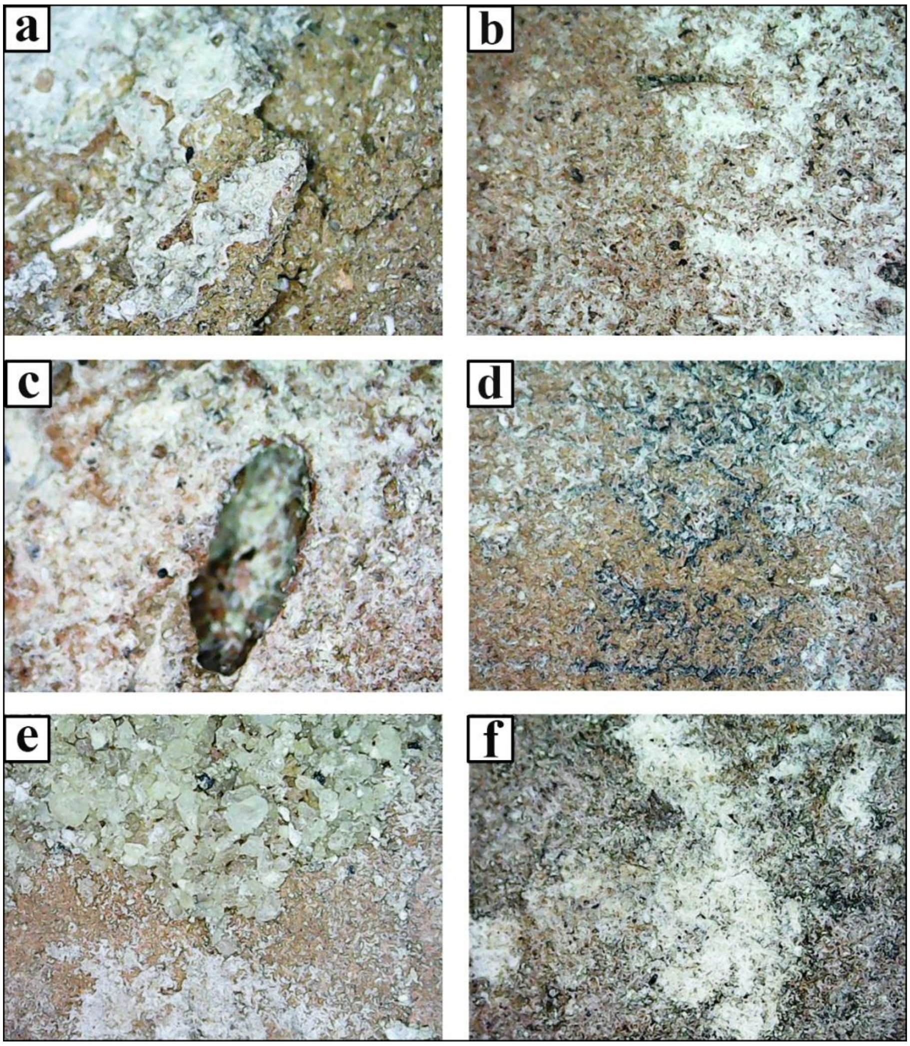

Figure 3. Shows the deterioration present on the potsherds. (a, b) sample P1, (a) fragility of the pottery body, (b) salts covering the outer surface. (c, d) sample P2, (c) loss in the body, (d) black spots on the surface. (e, f) sample P3, (e) a heavy accumulation of sand and dust, (f) salt and dust spots.

3.3. POLARIZED MICROSCOPY

Polarized microscopy (PLM) examination clarifies additive materials, the firing temperature, and the microstructure [36]. This method allows for the detection of coarse and small particles and the estimation of their quantity and grain size distribution [37]. Additionally, this examination was used to determine the texture of the pottery material [38]. Images of the potsherds by a polarized microscope are given in Figure 4. It is shown through the examination that the sample with code P1 contains quartz (Q), which is regarded as one of the sample's most essential components. Furthermore, the presence of calcite (C) and pottery powder (Grog-G) are used as fillers to improve the clay properties [39]. In addition, the coarse calcite grains seen in pottery may be natural components of the raw material or may have been introduced by the potters [37] (Fig. 4 a, b).

Sample P2 illustrates the presence of plagioclase feldspar (Pl) and various grain sizes and forms of quartz in a ground rich with iron oxides. In addition to the presence of the grog, as well as showing the boundary between the slip layer and the pottery body [40] (Fig. 4 c, d). Sample P3 shows plagioclase feldspar, calcite, and quartz. This sample is rich in iron oxides of a reddish-brown color [41]. Besides, the red slip was applied to large batches of pottery from the Old Kingdom, then polished to an intense shine [42]. The polarized microscope examination showed fine fabric in the first and third pottery samples. Additionally, the coarse texture appears in the second sample. These results indicate a variation in the method of pottery manufacturing at this archaeological site [30]. The type of clay, the forming, the firing process, and the surface treatment affect the texture of the potsherds [34]. Because these potsherds contain quartz, feldspar, pyroxene, mica, and red iron oxides, the examination results indicate that Nile clay was used in their manufacture [43]. Lower Egyptian Old Kingdom pottery is typically made of local Nile clay [32] (Fig. 4 e, f).

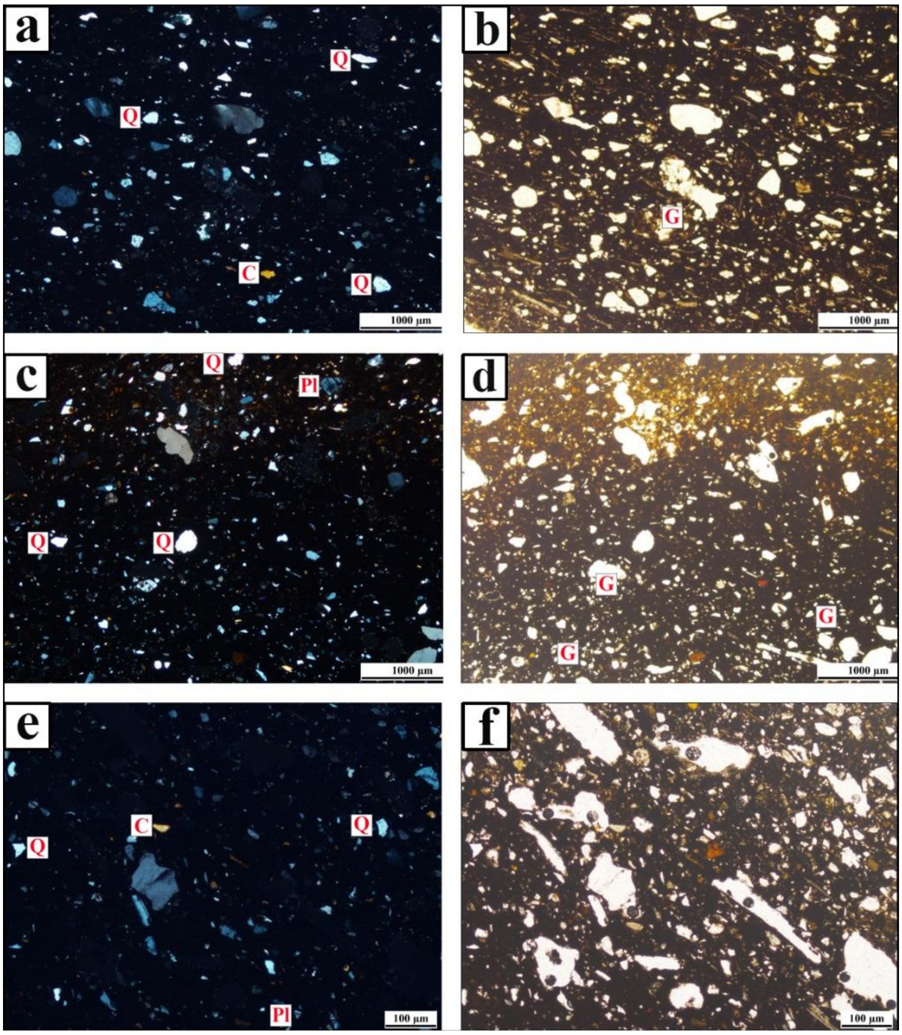

Figure 4. Shows the results of the polarized microscope. (a, b) sample P1, (c, d) sample P2, (e, f) sample P3. (a, c, e) P.L., (b, d, f) C.N .

3.4. SCANNING ELECTRON MICROSCOPY WITH EDX

SEM/EDX can determine the chemical structure of the potsherds. This method of investigation is helpful for archaeological study because it is speedy and non-destructive [44]. The images are shown in (Fig. 5). The sample p1 shows the presence of many cracks of relatively large size extending from the outer surface to the inner body layers because of soil pressure on the sherds for an extended period. Additionally, the SEM micrograph revealed homogeneity in the size of the granules [39]. The examination also shows great fragility in the pottery body and erosion of the grains because of exposure to the crystallization of salts. It also shows that the presence of pits, the peeling of the slip layer, and the filling of these pits with dust and sand causes deformation of the outer surface (Fig. 5a).

Table 1. Shows the concentration of elements [Wt. %] in potsherd samples.

Sample code	Elements										
	Fe	**Ca**	**K**	**Cl**	**S**	**Si**	**Al**	**Mg**	**Na**	**O**	**C**
P1	3.45	11.31	0.47	21.15	0.69	14.21	5.31	1.17	12.56	27.03	2.65
P2	4.86	17.44	1.54	0.29	13.47	25.37	5.42	1.32	1.09	26.18	3.02
P3	7.94	10.23	2.14	0.84	1.45	25.18	13.51	1.93	1.69	29.94	5.15

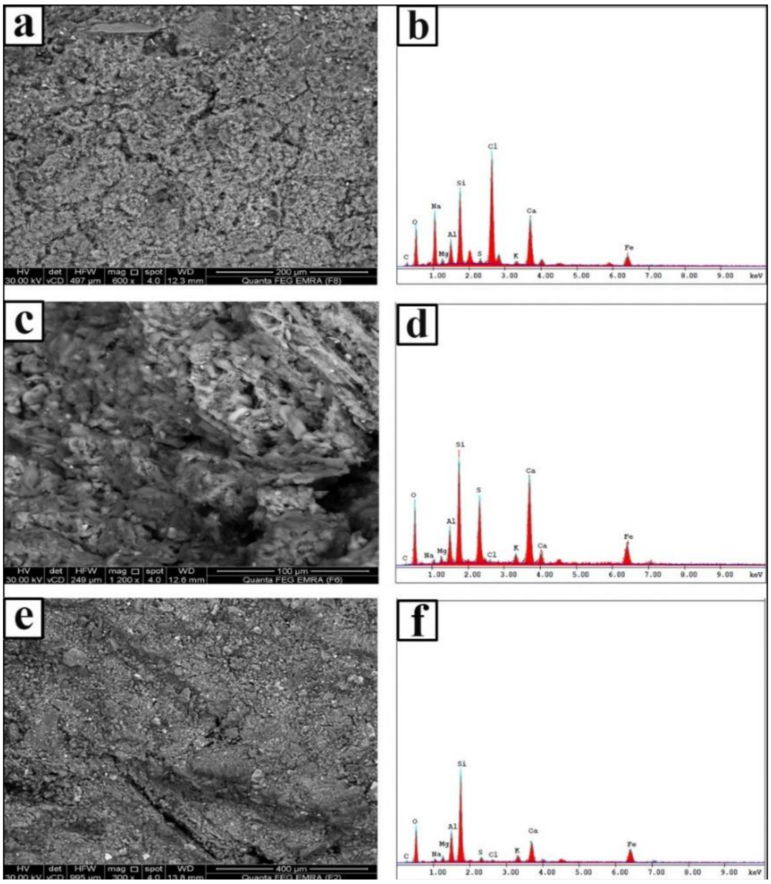

Figure 5. SEM images and EDX spectra of potsherds samples. (a, b) sample P1, (c, d) sample P2, (e, f) sample P3.

The sample P2 revealed heterogeneity in the size of the grains. Additionally, gaps are scattered over the body and filled with thick dust that sticks to them. In addition to the severe fragility, these sherds suffer from the expansion of the size of the pores, which helps to crystallize the salts on the outer surface and granulation, where the increase in the size of the internal pores causes an increase in the weakness and fragility of the pottery body [45] (Fig.

5c). The sample P3 shows the roughness of the outer surface of these sherds because of defects during manufacture. It also showed surface treatment using the slip layer [40]. Besides, the examination shows the presence of large cracks spread from the outer surface to the different body layers (Fig. 5e).

EDX was used to define the basic structure of each sample [46]. The type of clay used can be determined using SEM/EDX [47]. EDX determined the compositions of the potsherds, and the obtained data is registered in Table 1. The results show that sample P1 has a high percentage of chloride salts, especially sodium chloride salt, because of the burial of those sherds in soil rich in these salts (Fig. 5b). As for sample P2, the ratio of calcium oxide is also high due to the use of calcium carbonate as one of the additives for the clay. However, the presence of calcium oxide aids in the improvement of its physical qualities, resulting in a higher-grade product [48]. In addition, the percentage of chloride salts increases because of burial in soil rich in these salts (Fig. 5d). Finally, sample P3 has a high percentage of iron and aluminium oxide as one of the main components in the clay used to manufacture these sherds. The analysis results revealed a high percentage of magnesium oxide, which indicates that the clay used in the manufacture consists of montmorillonite [49] (Fig. 5f).

3.5. X-RAY DIFFRACTION

Mineralogical analysis acquired by XRD of the archaeological pottery can provide helpful information about raw materials, define the firing temperature, and identify deterioration aspects [51]. Besides, X-ray diffraction allows us to examine crystalline phases [48]. The XRD results of the analysis samples are obtainable in (Fig. 3). The XRD pattern of sample P1 contains quartz (34.4), albite (29.1), diopside (15.7), muscovite (15.5), and magnesium sulfate (5.4) (Fig. 6a). Quartz is an essential ingredient in this pottery sample. Albite is one of the plagioclase feldspars and one of the main components in the sample [33-50]. Diopside is formed by the reaction between clay minerals and calcium carbonate and starts at almost 850 °C [51]. Moreover, the temperature rise caused calcite's disappearance and diopside's presence between 850-900 °C [52]. Muscovite is an essential compound in the sample. Magnesium sulfate is a soluble salt because of burial in the soil containing those salts.

Sample P2 consists of quartz (38.4), orthoclase (32.4), magnesite (10.8), spinel (10.2), and hematite (8.2) (Fig. 6b). Quartz is an essential component of the pottery sample. Orthoclase is considered one of the primary components present in the sample. Besides, it is one of the feldspars among the clay components used in pottery making. Orthoclase vanishes at temperatures above 800 °C, which indicates that the burning temperature is higher than 800 °C [33]. Magnesite is one of the insoluble salts. Spinel appears at a temperature of 850–950 ^{o}C, indicating a high firing temperature of the sample. Hematite represents the red wash layer that was implemented on the outer surface. The color of potsherds depends on the chemical case of iron oxide, which is completely oxidized in the state of hematite [53]. Besides, the red slip layer resulting from the use of hematite distinguishes the pottery of the Old Kingdom [7]. XRD revealed the presence of quartz, hematite, and orthoclase in this sample, indicating that it was fired at least 900°C [54]. Additionally, the results of the XRD show that this sample is considered high quality due to containing spinel, which appears above 850 ^{o}C [30]. Analyzing X-ray diffraction spectra have been confirmed the existence of quartz; it is one of the most abundant minerals in potsherds. Besides, hematite is also present, giving this material its reddish color [55].

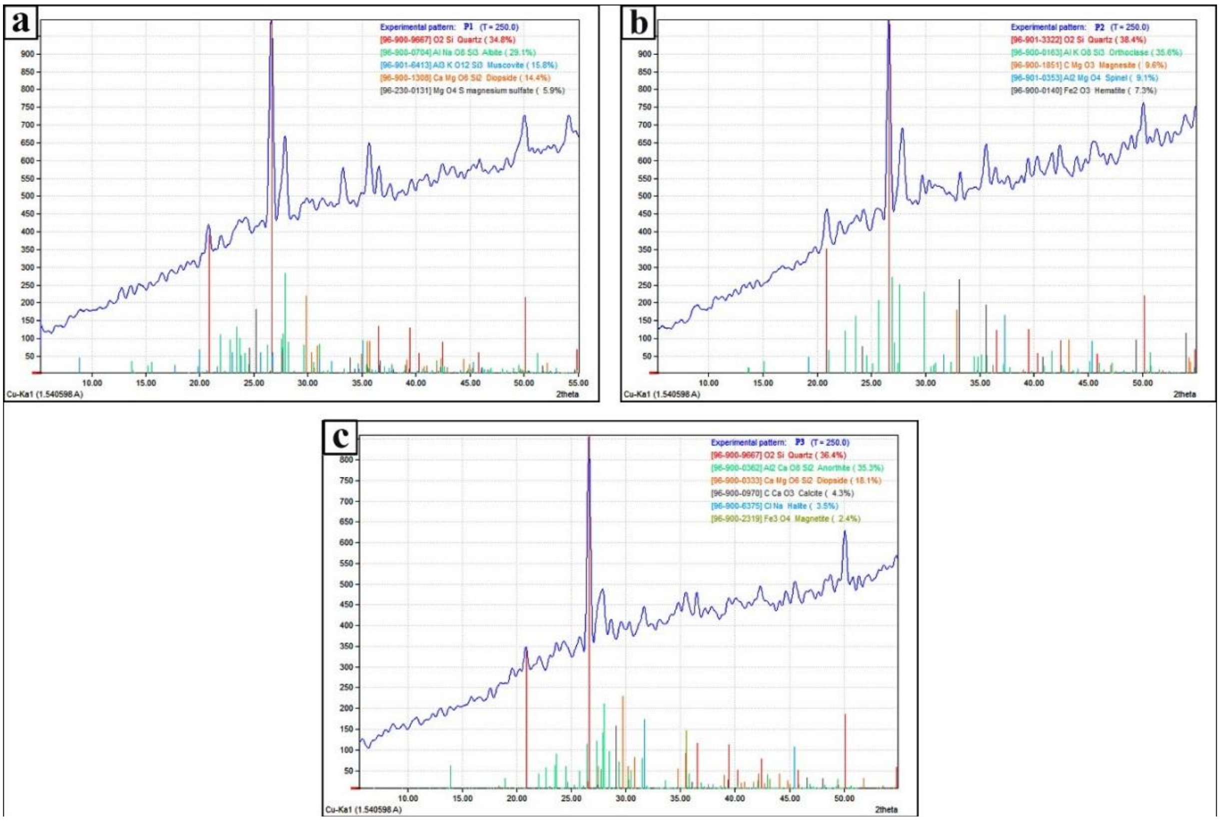

Figure 6. XRD patterns of the potsherds samples. (a) Sample P1, (b) sample P2, (c) sample P3.

Sample P3 consists of quartz (36.4), anorthite (33.2), diopside (18), halite (5.9), calcite (3.8), and magnetite (2.7) (Fig. 6c). Analyzing X-ray diffraction confirms the existence of quartz; it is one of the most abundant minerals in potsherds [41]. Anorthite is one of the plagioclase feldspars associated with clay minerals. Diopside is shaped at a high temperature, which provides us with a signal of the temperature of the firing of the sample [39]. Halite is a soluble salt resulting from the effect of the burial environment. The presence of calcite in pottery helps to identify the burning temperature. Calcite decomposition begins above 650 °C and fades above 800 °C [33]. Identifying calcite formations provides information on the raw materials used, the fire temperature, and the pottery's manufacturing process [37, 41]. Magnetite expresses iron oxides that are abundant in the core of the body.

The results of XRD show that these samples have the presence of a high ratio of quartz as a part of the composition of the potsherds [55]. The quartz found in the potsherds has a dual source: it could have been added as a filler, or it could have been created through the firing process from the decomposition of the clay silicates [51]. The mineralogical composition of salts and potsherds was determined by powder X-ray diffraction.

4. CONCLUSIONS

Various aspects of deterioration affect the internal structure of archaeological pottery buried in the soil. As a result, new methods of investigation and analysis were required to determine these potsherds' mineral and chemical content. Additionally, determine the aspects of deterioration that cause the fragility of potsherds. Throughout the research, it became clear that only a multi-technique approach could provide a thorough description of complicated materials like pottery. On the other hand, using a single method may produce unfinished and misleading results in some circumstances. For example, digital and scanning electron microscopy were employed to observe textural and microstructural features. They revealed salt crystallization, accumulation of dust and sand on the surface, black spots, cracks and fragility in the pottery body. According to PLM's findings, Nile clay was employed in the production of potsherds. The potsherds were also subjected to SEM/EDX analysis, and the results revealed that the clay employed in their manufacturing consists of montmorillonite. Regarding XRD analysis, results show that the firing temperature of the potsherds was about 850-950 ºC. The presence of iron oxides, primarily hematite, gives the potsherds their red color. The pottery has very high toughness. From morphological and mineralogical studies, can be concluded that the temperature reached was high enough for diopside and spinel formation.

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