Examination, Analysis and Conservation of Some Archaeological Pottery Vessels from Saqqara Excavation

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Abstract

This study deals with one of the important topics related to the conservation of excavated pottery. Due to their presence in various excavation sites and the great importance in the history of many archaeological sites. Therefore, it was necessary to use different methods of examination and analysis to identify the damage of pottery, in addition to applying proper conservation methods to the selected objects. A visual examination and documentation by AutoCAD were used to clarify the deterioration, such as the spread of salt and dust on the surface and the presence of missing parts. USB digital microscope, polarizing microscope, SEM-EDX and XRD were applied to identify the chemical and mineralogical composition of these objects. The results of the investigation showed that the selected pottery suffered from black spots, different cracks, high porosity, and heterogeneity in the grains size. The results of the polarized microscope investigation revealed the presence of quartz, calcite, pottery powder (grog), and plagioclase feldspar mainly Anorthite. SEM-EDX analysis showed that a high percentage of aluminum oxide and silica as the main components in the clay used in pottery making. XRD analysis revealed that the samples consist mainly of Quartz, Diopside, Illite, Muscovite, Orthoclase, Anorthite, and Hematite that are the main components in the manufacture of these objects. The treatment included mechanical and chemical cleaning. Paraloid B72 was used for joining one of the pots at 60% in acetone. Additionally, the missing parts were completed with a paste of pottery powder, dental gypsum, and primal AC33. Besides, Paraloid B72 consolidated these objects at 5 %.

Key words:

pottery, deterioration, conservation, XRD, SEM-EDX.

الملخص

تتناول هذه الدراسة أحد الموضوعات المهمة المتعلقة بعلاج وصيانة الآثار الفخارية المستخرجة من الحفائر، وذلك لكثرة وجودها في مواقع الحفائر المختلفة، وما تمثله من أهمية بالغة في تأريخ العديد من المواقع الأثرية. لذلك كان من الضروري استخدام طرق مختلفة للفحص والتحليل لتحديد مظاهر التلف المختلفة التي تعاني منها الأواني الفخارية محل الدراسة، بالإضافة لتطبيق طرق الحفظ المناسبة على القطع المختارة. تم استخدام الفحص البصري والتوثيق بواسطة الأوتوكاد AutoCADلتوضيح مظاهر التلف المختلفة مثل انتشار الأملاح والأتربة على السطح وكذلك وجود أجزاء مفقودة في بعض الأجزاء من البدن. بعد ذلك تم استخدام الفحص بالميكروسكوب الرقميUSB digital microscope ، والميكروسكوب المستقطب PLM، والميكروسكوب الإلكتروني الماسح المزود بوحدة تحليل عنصري (SEM-EDX) والتحليل بحيود الأشعة السينية (XRD)، لتحديد التركيب الكيميائي والمعدني لهذه الأواني. وقد أظهرت نتائج الفحص أن

هذه الأواني تعاني من وجود بقع سوداء منتشرة على السطح وكذلك وجود شروخ مختلفة الأحجام وعدم التجانس في حجم الحبيبات. أما بالنسبة للميكروسكوب المستقطب فقد أظهرت النتائج وجود معادن الكوارتز والكالسيت ومسحوق الفخار وأحد معادن الفلسبارات البلاجيوكليزية مثل الانورثيت. في حين اظهر تحليل SEM-EDX ارتفاع نسبة أكسيد الألمونيوم والسيليكا كأحد المكونات الأساسية في الطفلة المستخدمة في صناعة تلك الأواني الفخار. وقد بين تحليل XRD أن العينات تتكون بشكل أساسي من الكوارتز، الدايبوسيد، الإيليت، المسكوفيت، الأورثوكليز، الأنورثيت والهيماتيت التي تعتبر من المكونات الأساسية الداخلة في صناعة هذه الأواني. وقد تمَّ إجراء عمليات العلاج والصيانة عن طريق التنظيف الميكانيكي والكيميائي، ثم إجراء عمليه التجميع لاحد الأواني باستخدام البارالويد ب-٧٢ بتركيز ٦٠%، بعد ذلك تم استكمال الأجزاء المفقودة بمسحوق الفخار مع جبس الأسنان والبريمال Ac33 بتركيز ١٥%، وفي النهاية تم تقوية تلك الأواني بالبارالويد ب-٧٢ بتركيز ٥%.

الكلمات المفتاحية:
فخار، تلف، صيانة، حيود الأشعة السينية، الميكروسكوب الإلكتروني الماسح.

1. Introduction

Archaeological pottery buried in the soil is exposed to serious deterioration because of soil pressure (Smith, 1998). Although pottery is a material with high resistance to chemical damage, it is less resistant to mechanical damage, especially when buried in the soil, where high-firing pottery shows higher resistance to mechanical loading than low-firing pottery (Buys and Oakley, 1993). Water can cause a physical and chemical deterioration to pottery, where it catalyzes many other damage factors, and thus facilitates chemical reactions (Mladen, 2011; Cronyn, 1993).

Ground water and moisture present in the burial environment also have bad effects on poor-burning pottery as they can cause severe deformation and damage such as the erosion of some parts of the body (Jones, 2008). Moisture is considered one of the factors that cause deterioration to the monuments, and since pottery is a porous material, this helps to increase the moisture content of the pottery through the capillary property (Madkour and Khallaf, 2012). The evaporation of salt-bearing water is one of the most important factors causing the speed of salt crystallization, whether on the outer surface or between the internal pores that cause fragility and weakness of the body components (Montana, 2013).

Pottery objects are affected by salt solutions, as the crystallization of salts on the outer surface leads to deformation of the drawings and colors (Faulding and Thomas, 2000). The salts increase the mechanical stresses because of their crystallization in the internal pores of the body, which ultimately leads to flaking and loss of parts of the surface (Costa Pessoa et al, 1996; Beskhyroun et al, 2019). Additionally, the increase of salts rate in the internal pores leads to the presence of internal pressures that cause weakness and cracks (Hemeda, 2018; Stryszewska and Kanka, 2017). The crystallization of the salts causes the formation of solid crusts on the surface as well as between the layers of the pottery body. It leads to the disintegration of some parts of the pottery objects (El-Badry, 2019).

The external shape of salt crystals depends on the nature and type of salt and the degree of concentration of these solutions as well as the changes in the rates of temperature and relative humidity in the external atmosphere (Rodriguez and Doehne, 1999). This study focuses on the analysis and conservation of some pottery vessels discovered in the excavation of the Faculty of Archeology, Cairo University in Saqqara. The excavation mission was under the supervision of Prof. Dr. Ola El-Augizi during the 2018-2019 season. The discovered objects are dated to the New Kingdom. This paper aims to identify the chemical and mineral compositions of the studied pottery vessels using different examination and analytical methods. It was applied to determine the condition of the objects and to apply proper conservation methods to the selected pottery.

2. Materials and Methods

2.1. Materials

Two pottery vessels were selected from the excavated pottery to apply the study. These objects suffer from changed aspects of damage. Therefore, different examination and analysis methods were used to identify the causes of damage to pottery vessels. The pottery vessels were coded to facilitate the discussion as shown in (Table 1).

Table (1) shows the description of the pottery vessels.

Code	Vessels Description
A1	It consists of four parts of pot sherds. It has various aspects of damage such as weakness in the body and flaking of the slip layer. There are some manufacturing defects in various parts of the body and crystallization of salts.
A2	It is a cylindrical vessel with a loss in the body from the neck. It suffers from the accumulation of sand and dust on the surface.

2.2. Methods of Examination and Analysis

2.2.1. Visual Examination

This examination is one of the primary and important way to identify the problems that present on the pottery vessels. Through the visual examination, it is possible to identify the state of the object and make a preliminary assessment of its condition.

2.2.2. Documentation

AutoCAD version 2018 was used to record the dimensions of the object and clarify the various types of damage that exist on the pottery vessels.

2.2.3. USB Digital Microscope

USB 2.0 interface, Linux, Mac OS & above 10.5.5, from (10X-500X), Model: PZ01.

2.2.4. Polarized Microscope (PLM)

Type of polarized microscope: Nikon ECLIPSE LV100POL (DS-FI1) Made in Japan. The used PLM is available at Department of Geology, Faculty of Science, Cairo University, Egypt.

2.2.5. Scanning Electron Microscope with Elemental Analysis Unit (SEM-EDX)

SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analysis), was used in this research. Specifications: accelerating voltage 30 K.V., magnification14x up to 1000000 and resolutions for Gun.1n), FEI Company, Netherlands. The used SEM-EDX is available at the General Authority for Mineral Resources in Dokki – Egypt.

2.2.6. Mineralogical Analysis by XRD

Type of X-ray analysis device used: Diffractometer-PW 1480- 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. The XRD unit used in this work is present at the Faculty of Archeology, Cairo University, Egypt.

3. Results and Discussion

3.1. Visual Examination

The main aim of this stage is to diagnose the various aspects of damage. These pots suffer from flaking, fragility, and weakness in the outer slip layer. As well as the presence of missing parts and pits of various sizes, which are filled with dirt and sand. Besides, vessel A1 is broken to several parts because of soil pressure (fig. 1).

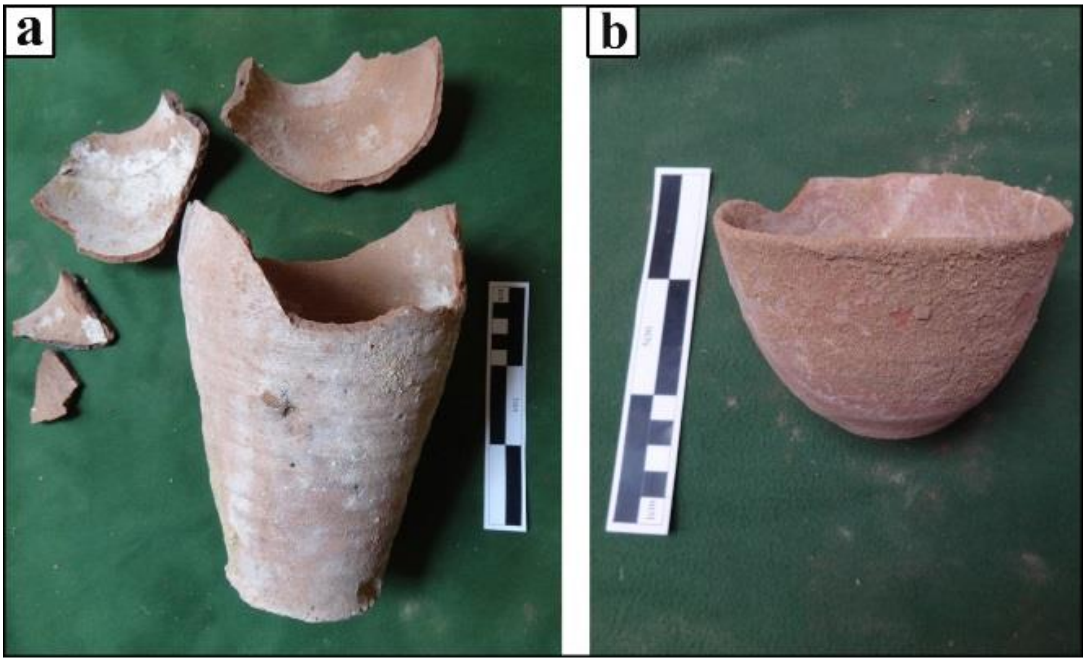

Fig.1 shows the pottery objects understudy. (a) A1, (b) A2.

3.2. Documentation

AutoCAD program was used to clarify and document the dimensions of the pottery objects. Additionally, recording the various aspects of damage such as the presence of salts, dust, and sand that spread on the surface (fig. 2).

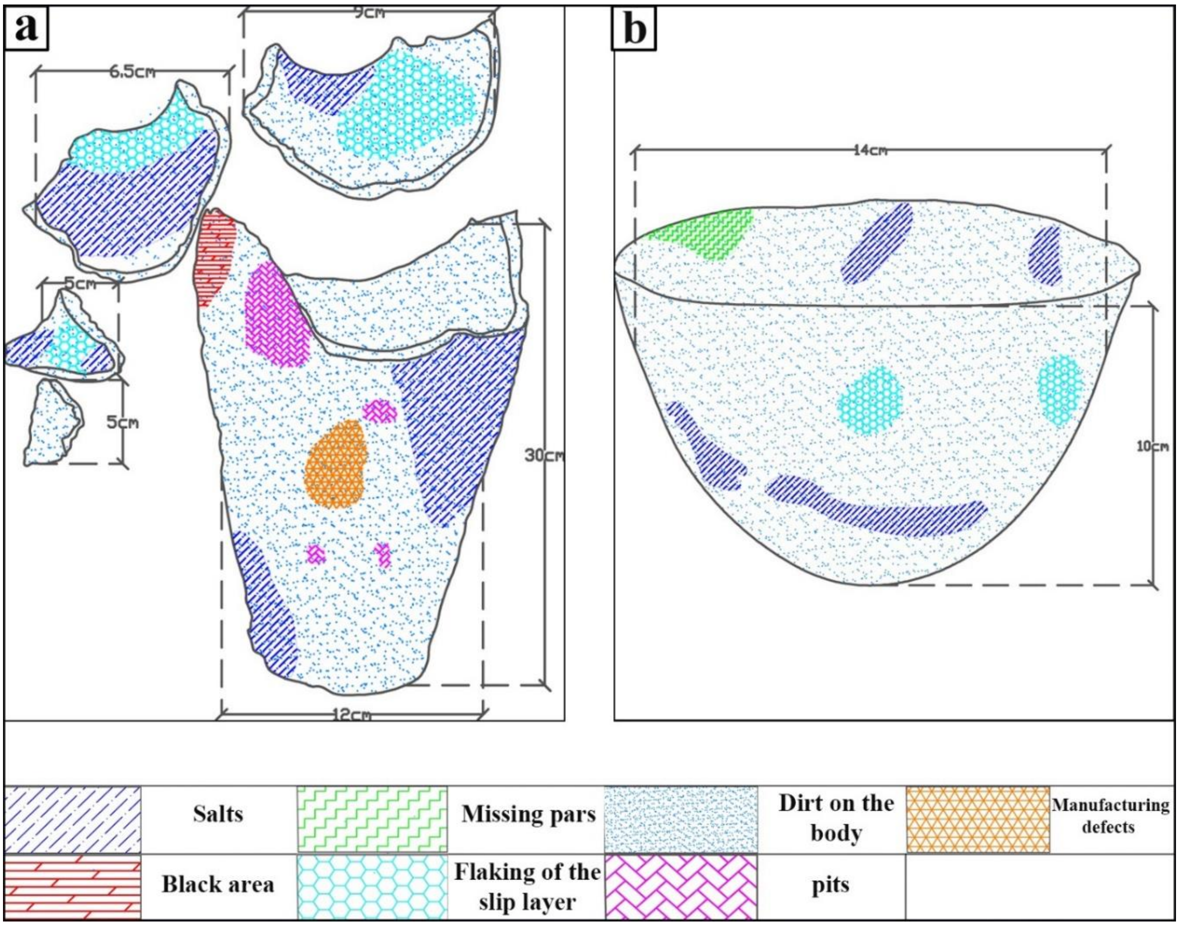

Fig. 2 shows the documentation of the different aspects of pottery deterioration by AutoCAD. (a) A1, (b) A2.

3.3. USB Digital Microscope

This microscope was used to examine the pottery with (10X - 100X) to identify the different aspects of deterioration that can't be seen with the naked eye (Dawi et al, 2019) such as crystallization of salts, color change, black spots, and cracks of various sizes (Ibrahim and Mohamed, 2019) (fig. 3,4).

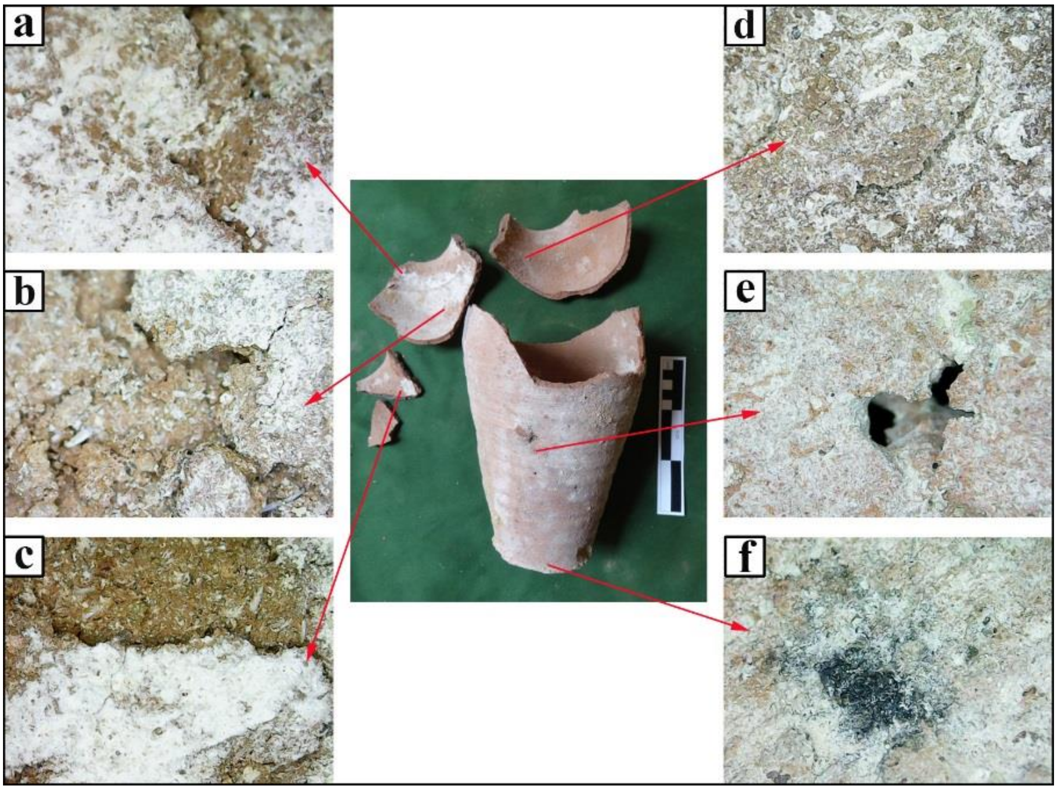

Fig.3 shows the damage present on the pottery object with code A1. (a) a crack extending from the slip layer to the depth of the body, (b) fragility in the inner slip layer, (c) cracks, separations of the inner slip and salts, (d) salt crystallization and separation of parts from the body, (e) pits and holes, (f) black spots on the body.

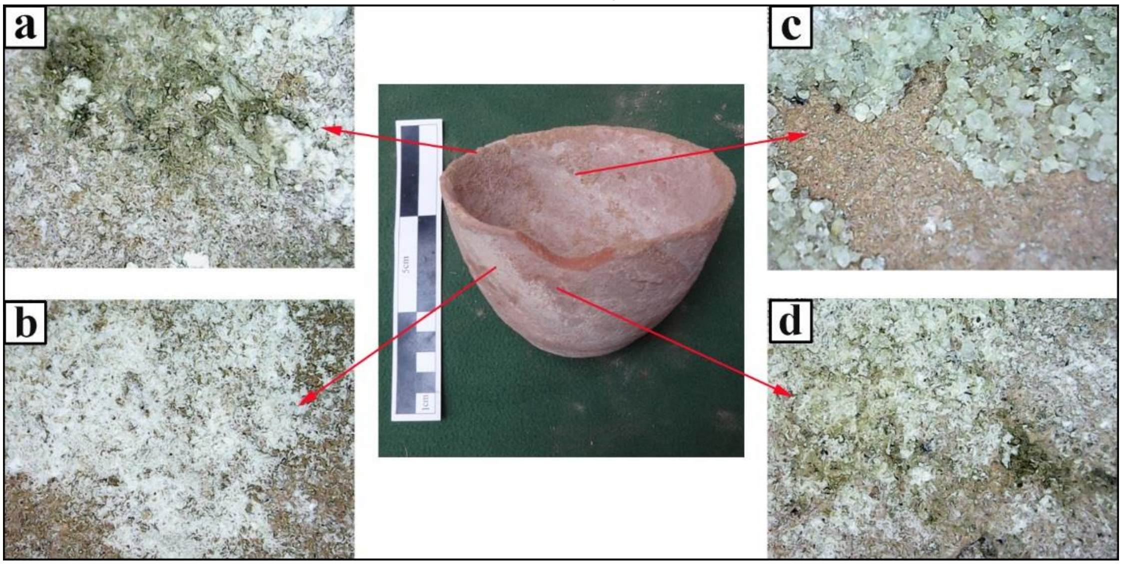

Fig.4 shows the damage present on the pottery object with code A2. (a) Dirt on the surface, (b) salt covering the surface, (c) grains of sand (d) chromatic change.

3.4. Polarizing Microscope

It is shown through examination that sample A1 consists of quartz (Q), which is one of the basic components of the sample. Besides, the presence of calcite (C) and pottery powder (Grog-G), which were used as a filler to improve the clay properties. Sample A2 shows the presence of the plagioclase feldspar, which takes the form of twinning, which proves that the source of the raw clay is from Nile clay (Wodzińska, 2009). The presence of the plagioclase crystals in the sample indicates an increase in firing temperatures above 800 ◦C (López-Arce et al, 2003). Additionally, the presence of different grain sizes and shapes of quartz in a ground that is rich with iron oxides (Balliranoa et al, 2014) (fig. 5).

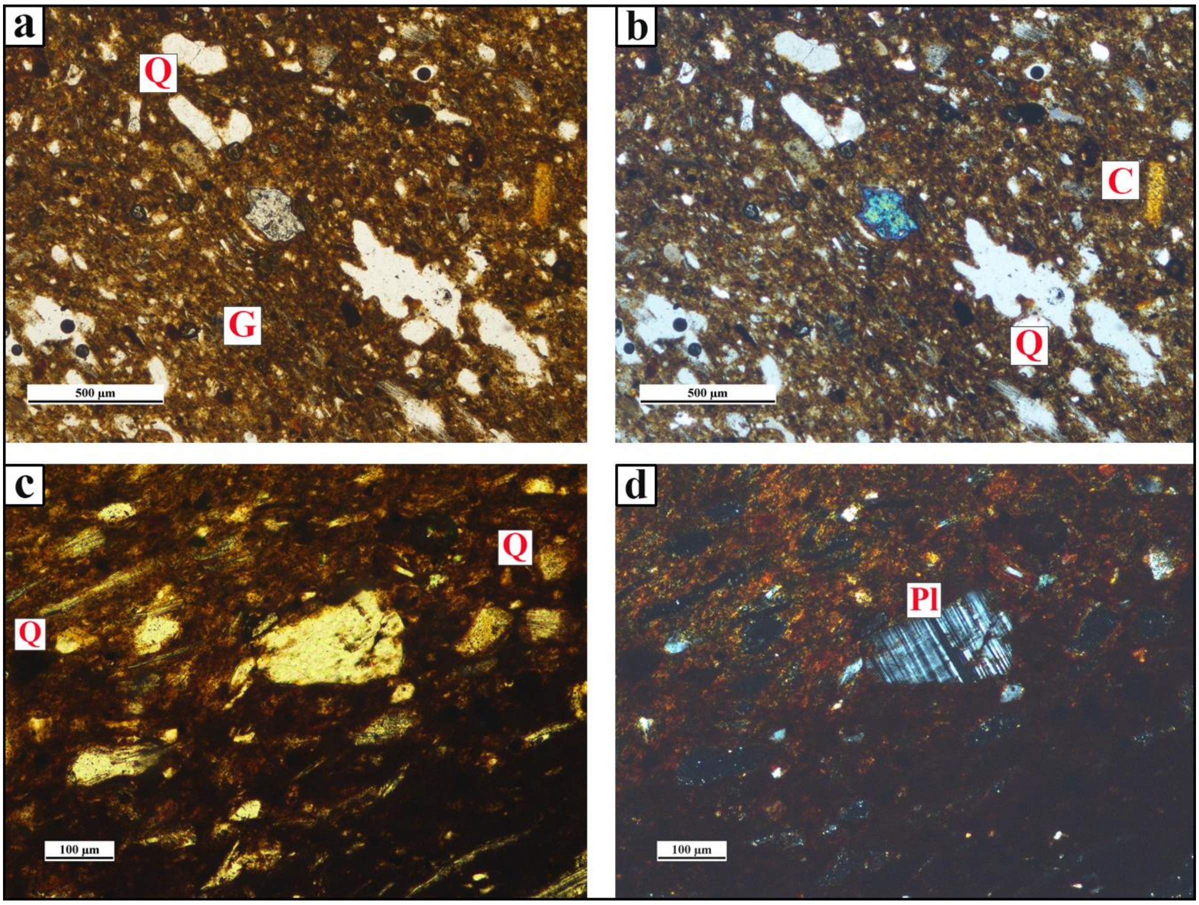

Fig. 5 shows the results of the polarized microscope's examination. (a, b) A1, (c, d) A2, (a, c) P.L., (b, d) C.N.

3.5. Scanning Electron Microscope with Elemental Analysis Unit (SEM-EDX)

3.5.1. The Examination with Scanning Electron Microscope (SEM)

The SEM micrograph of sample A1 revealed the heterogeneity in the size of granules. Additionally, the sample suffers from severe fragility, as well as the presence of pits that filled with dust and sand. Sample A2 shows a thick accumulation of sand on the outer surface. Sand was tightly adhered to surface due to its incorporating with the salt crystals that are widely spread on it. Besides, the abundance of dust on the surface due to burial in the soil for a long period, which resulting in deformation of the surface and covering the slip layer, (fig. 6)

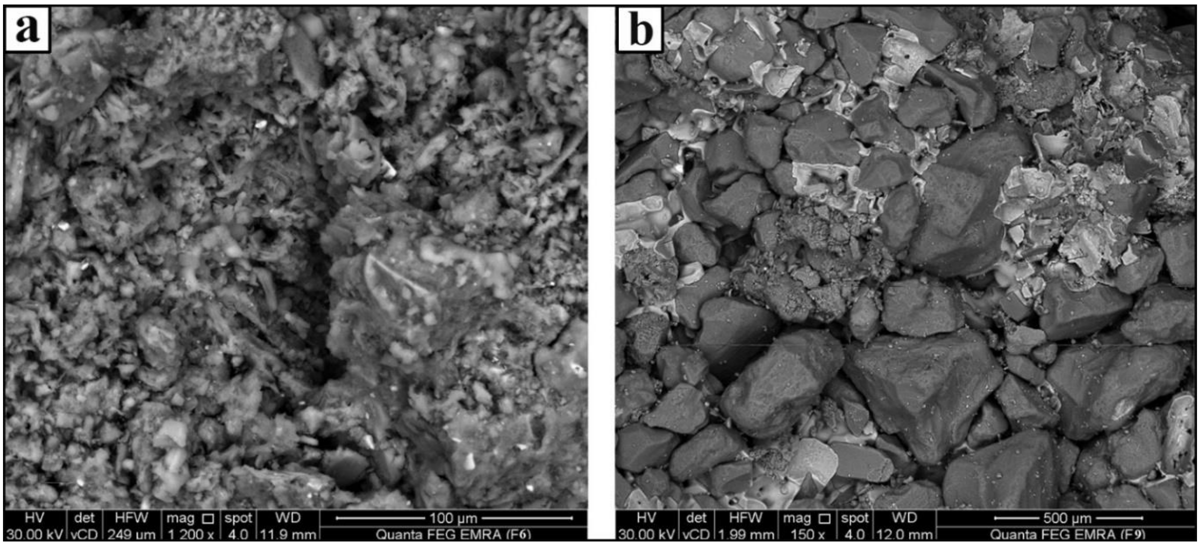

Fig. 6 shows the results of the SEM investigation of pottery samples; (a) represents the heterogeneity of the particle size in sample A1, (b) represents the accumulation of sand on the surface in sample A2.

3.5.2. The Analysis with Scanning Electron Microscope with EDX

The result of SEM-EDX analysis indicates that sample A1 has a high percentage of iron oxide, which is considered one of the main components of the clay. In addition to increasing the percentage of aluminum oxide and silica as the basic components of the clay. It was observed through analysis the presence of a high percentage of magnesium oxide, which indicates that the clay consists of montmorillonite (Elghareb, 2019). In addition to the high percentage of calcite due to the use of calcium carbonate in the slip layer. Sample A2 shows the high ratio of chloride salts, especially sodium chloride, which may have migrated from the surrounding soil. (Madkour and Khallaf, 2012) (fig. 7, 8).

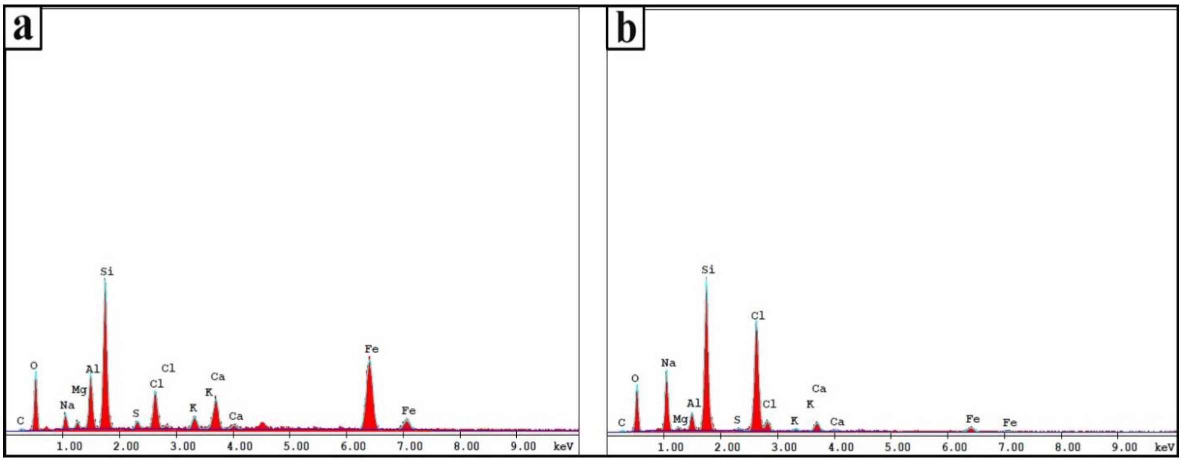

Fig. 7 shows the results of the SEM – EDX; (a) A1, (b) A2.

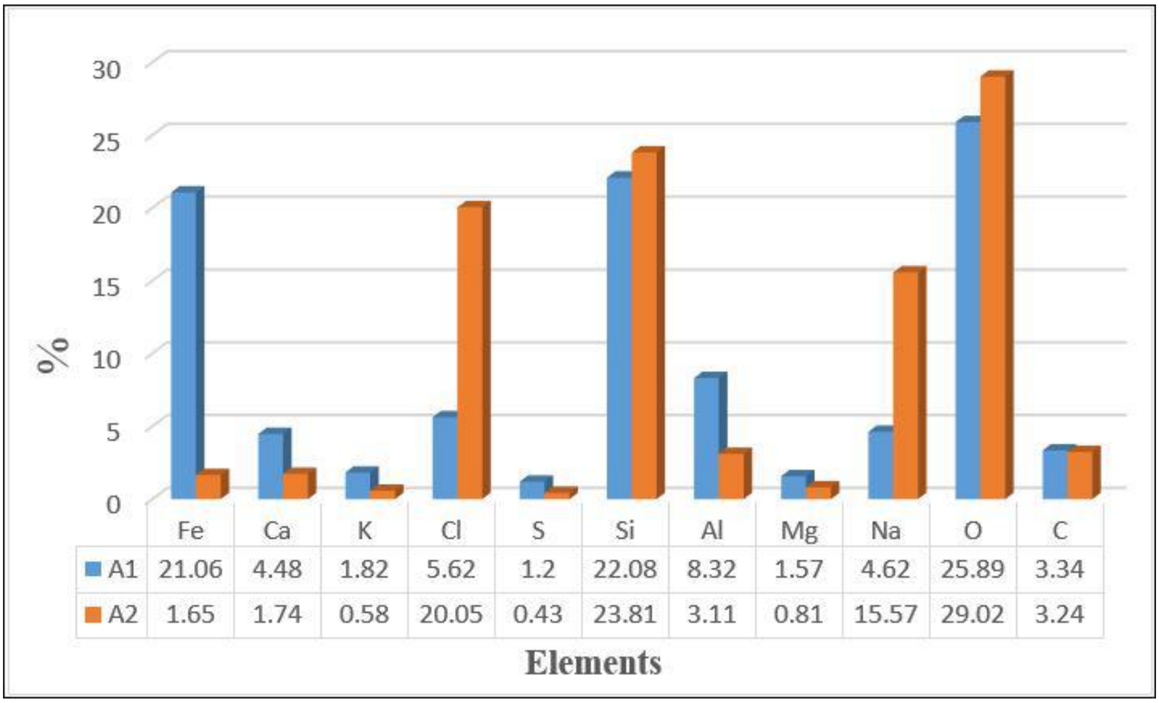

Fig. 8 shows concentration of elements (%) of pottery samples

3.6. X-Ray Diffraction Analysis

The results of XRD show that Sample A1 is composed of quartz (SiO_2) as one of the basic components associated with clay, which is used in pottery making or added to clay as a filler and it affects the plasticity of the used clay (Ibrahim and Mohamed, 2019). Orthoclase ($KAlSi_3O_8$) is a basic component present in the sample. Diopside ($CaMgSi_2O_6$) is a result of the transformation of carbonates and indicates that the firing temperature is about 850 °C as it is formed at high temperatures (Cultrone et al, 2001). The low percentage of calcite ($CaCO_3$) was detected in the studied sample by XRD. The presence of calcite particles that decompose between 600-850 ^oC. might initially indicate that firing temperature is below 850 ^oC. It indicates to estimate the firing temperature between about 800-850 ^oC due to the presence of both diopside and calcite. High firing minerals such as ghehlenite, mullite, spinel, and cristobalite were not noticed by XRD analyses that support the above result of firing temperature estimation not above 850°C. (Al-Bashaireh et al, 2018).

Hematite (Fe_2O_3) represents the red slip layer that was carried out on the surface. Halite (NaCl) is a soluble salt resulting from the influence of the burial environment. Sample A2 consists of quartz (SiO_2) that is a main component of the sample. Halite (NaCl) is a soluble salt resulting from the influence of the burial environment. Anorthite ($CaAl_2Si_2O_8$) is one of the plagioclase feldspar associated with clay minerals. Illite ($KAl_2Si_3.AlO_{10}(OH)_2$) is one of the components of the raw material from which the pots are made. Diopside ($CaMgSi_2O_6$) indicates the high temperature of pottery burning. The use of XRD helped us in the identification of pottery vessel components. Additionally, it shows the minerals transformation occurred during firing the clay, which indicated the burning temperature of pottery (Abdel Rahim, 2011) (fig. 9).

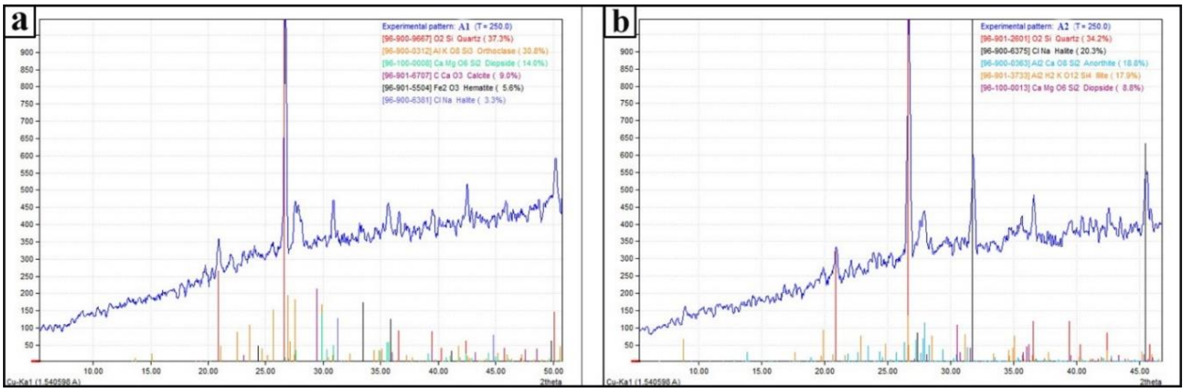

Fig. 9 shows the results of the XRD of pottery samples; (a) A1, (b) A2.

4. Treatment and conservation of the pottery vessels

4.1. Cleaning

It is necessary before the cleaning to identify the type of pottery, its mineral composition, and the nature of dirt on the surface. Cleaning means removing dirt and dust from the surface of the object, but it does not mean removing any materials that were stored inside it (Abd-Allah et al, 2010). Therefore, some investigations were necessary carried out to determine the chemical composition of the different aspects of deterioration to use the correct and effective methods of cleaning (Plenderleith, 1996).

4.1.1. Mechanical cleaning

The main aim of mechanical cleaning is to remove or reduce the solid materials adhering to the surface of the archaeological objects and to reduce the need for chemical cleaning (Faulding & Thomas, 2000). Where scalpels were used to remove the hard layers on the surface, in addition to use soft brushes to remove dust.

4.1.2. Chemical cleaning

Distilled water is used without any additives when starting the chemical cleaning then different solutions are used such as acetone to remove dirt and salts and alcohol to remove solid stains (Adrian & Paez, 2007). The degree of purity of the water should be known before it is used to clean archaeological pottery (Paterakis, 1996). Cotton poultices were used to remove the salts from the body of the pottery vessels by acetone diluted with distilled water in a ratio of 1: 1.

4.2. Joining

The joining process is considered as an important stage to join the broken parts together to recover the original shape of the object, and this can be called the process of reconstruction of the artifact. So, the reassembly of the artifact reverses its aesthetic value (Burnham & Tarling, 1975). At this stage, paraloid B-72 with a concentration of 60% in acetone was used to join the parts of the pottery body with code (A1) (Ibrahim et al, 2020).

4.3. Completion

Completion is one of the important restoration stages because this process helps us to prepare the object for museum display (Otabek, 2013). There were many missing parts in these pots. So, the intervention was required to complete these parts with appropriate completion materials.

According to a previous study, it was proved that the use of new pottery powder 70% + dental gypsum 30% + primal AC33 at 15% paste is considered a good mixture for completion of pottery objects. Hence, this paste having similar properties to archaeological pottery (Ibrahim and Mohamed, 2018). So, we applied the same paste to complete the missing parts.

4.4. Consolidation

Consolidated materials are used to improve the cohesion of archaeological objects (Pinto & Rodrigues, 2008). So, this process aims to improve the physical and mechanical properties of the treated objects (Jroundi et al, 2017). Therefore, Paraloid B-72 was used at 5% in acetone to consolidate these pottery vessels. This material gave good results in increasing the strength and cohesion of the pottery body. The different restoration stages of these vessels are shown in (fig 10, 11).

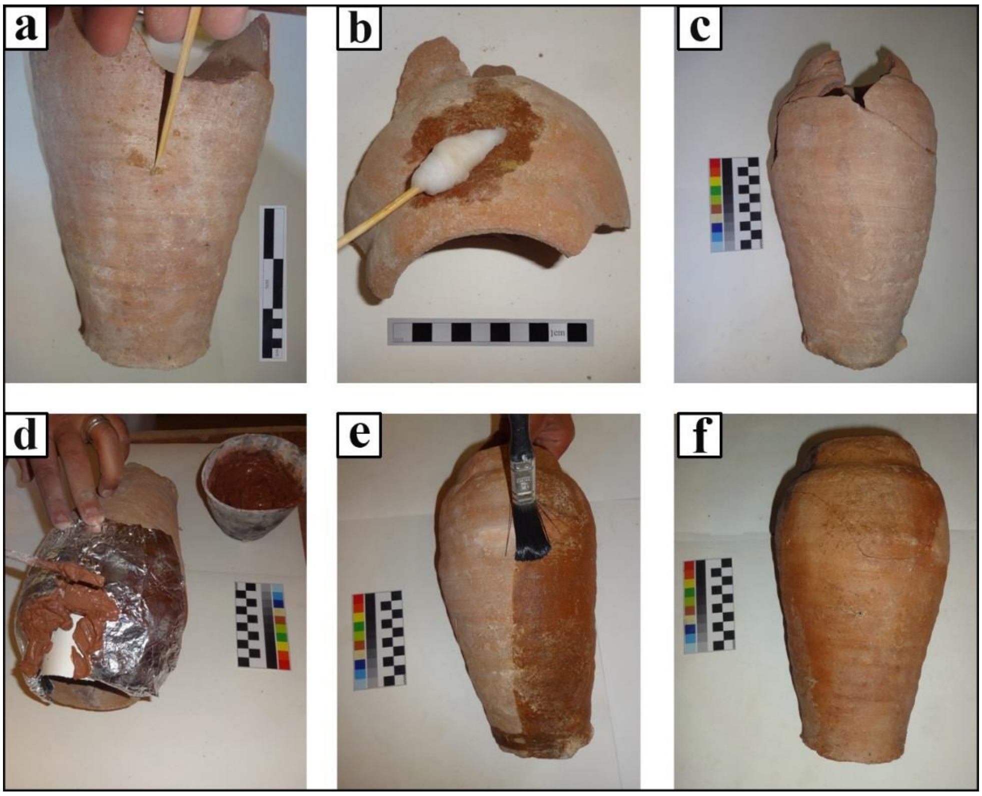

Fig. 10 shows the stages of treatment of vessels with code A1. (a) Mechanical cleaning to remove hard dirt from the vessel, (b) chemical cleaning with water and acetone 1:1, (c) after finishing the joining of the object's parts, (d) applying the completion materials, (E) applying the consolidation materials with a brush, (F) the pottery vessel after finishing of the restoration.

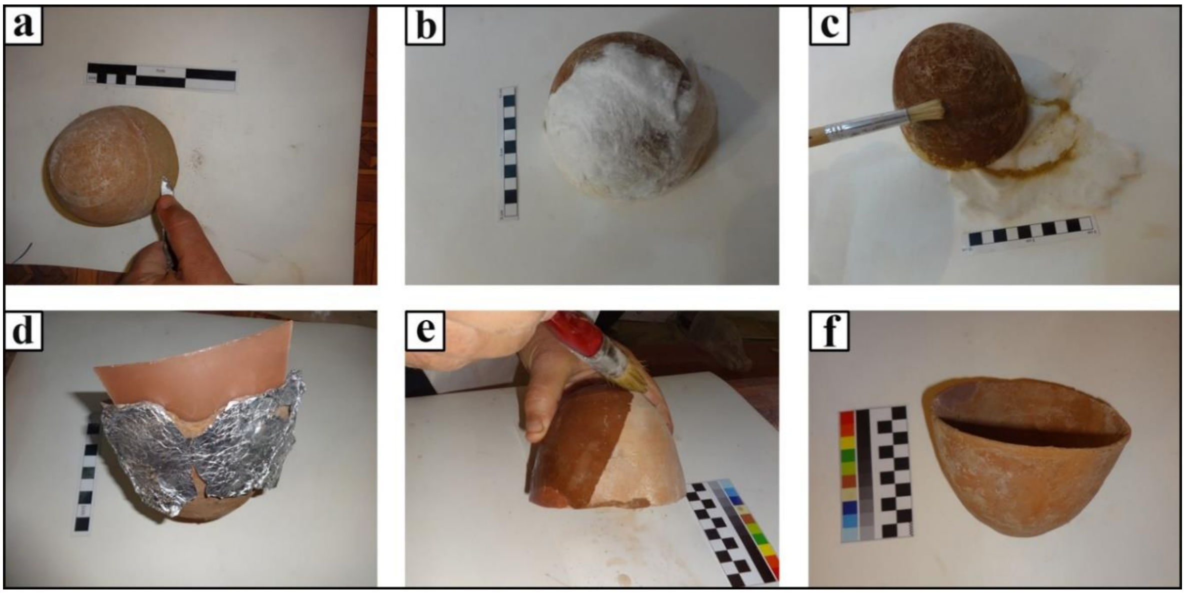

Fig. 11 shows the stages of treatment of vessel with code A2. (a) Mechanical cleaning, (b) use of cotton poultices to remove crystalline salts, (c) cleaning of salts, (d) securing the edges, and use of wax to facilitate completion, (e) application of consolidation materials, (f) the pottery vessel after finishing of the restoration process.

5. Conclusion

Archaeological pottery buried in the soil suffers from various aspects of deterioration that affect the internal structure. Therefore, it was necessary to use different methods of examination and analysis to identify the mineral and chemical compositions of these objects. Additionally, identify the aspects of damage that cause weakness and fragility of these pottery vessels. Visual examination and documentation by AutoCAD showed the presence of pits and holes of various sizes. Besides crystallization of salts that cause deformation of the outer surface. The USB and SEM micrograph showed the presence of some cracks spreading in the body, as well as the heterogeneity in the grains size. A polarized microscope revealed the presence of pottery powder (grog) and calcite as fillers to improve the properties of the clay used in pottery making. The results of SEM-EDX proved that the clay used in the making of vessels consisted of montmorillonite due to the high percentage of magnesium oxide. XRD analysis showed the presence of sodium chloride salt as one of the salts spread in the burial environment, in addition to the presence of quartz, which is the main component of clay. Besides, the presence of a diopside indicates that the firing temperature is about 850 °C. These results helped us to select the best materials for the treatment of pottery vessels. The use of acetone diluted with distilled water in a ratio of 1: 1 is considered one of the best materials that gave good results to remove dust and salts spread on the surface. Also, the use of Paraloid B-72 in either the joining or consolidation process had a positive effect. The mixture that consist of pottery powder with dental gypsum and primal Ac33 gave good results in completing the missing parts of the pottery body.

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