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Original article

IDENTIFICATION AND RESTORATION OF LATE ROMAN AMPHORA, 4^{TH}-6^{TH} CENTURIES AD. FROM EL-BAHNASA SITE, MINIA, EGYPT

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Abstract
Amphoras are large ceramic vessels which were used, in the Greaco-Roman period, to ship wine and other liquid products throughout the Mediterranean. An amphora was excavated at El-Bahnasa archaeological site at the south of Egypt and it dates back to the Late Roman period (4^{th}-6^{th} centuries AD.). The condition of the amphora was very poor and suffered from many deterioration phenomena including, accumulation of dirt and soil residues, narrow and wide cracks, decay and fragility of pottery body and crystallization of salts. Furthermore, many shards are broken from the body and some are missing. The aim of the present paper is to study the chemical and the mineralogical composition of the clay body, the soil residues and the crystallized salts and to restore it. Different analytical methods were used including; X-ray diffraction (XRD) and scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS). The results obtained by (XRD) reported that clay body contains Quartz (SiO_2), Calcite ($CaCO_3$), Anorthite ($CaAl_2Si_2O_8$), Halite (NaCl), Magnetite (Fe_3O_4), Dolomite (Ca $Mg(CO_3)$ and Hematite (Fe_2O_3), while the salt is Halite mineral (Sodium chloride). The soil residues sample consists of Halite, Quartz and Calcite. Different restoration treatments were carried out on the amphora comprising; mechanical and chemical cleaning, consolidation, bonding and replacement processes.

Keywords: *Roman, Amphora, El-Bahnasa, Deterioration, Accumulation, Wide cracks.*

1. Introduction
Commercial amphoras are large ceramic vessels that were used from 1500 BC. to 500 AD to ship wine and other products to the ancient Greek and Roman empires throughout the Mediterranean [1][2]. As the Roman Empire grew, so did the shipping of goods for military an trade, which required more packaging. Amphoras supplied the Empire and were especially important for bringing wine and other products back to Rome. Amphoras differ from other ceramic jars in that their fat bodies, ranging from egg to bottle shapes, were designed specifically for shipping a large quantity of liquids (wine, olives, oils, and processed fish sauce or dry products such as grain, nuts, and salted fish [1][3]. Greek and Roman clay shipping amphoras were difficult to throw away. They had to be thick-walled, as a protection against breakage and seepage during rough and dangerous sea voyages [4]. Amphoras were produced on a wheel in a multistep process with intermittent

drying to add rigidity. Some were produced as modular pieces and then joined. After forming the body and base, the vase was turned upright and supported by a chuck while the neck and rim were formed and the handles were attached last [1]. Unglazed ceramic pottery is porous, and so an impermeable lining was required for wine or other no viscous liquids. Resin made from pine trees or an interior coating of pitch was most often used as the barrier lining for wine amphoras [1][3]. Regarding of the shapes of amphoras, the main part of the jar was belly and the rim or lip at the top of the neck was an aid in controlling the flow of the liquid being poured. The main part was joined to the neck by a sloping shoulder and the bottom was tapered shape [4]. Throughout the Roman periods, the shapes from about 30 BC. on became more indicative of the contents than of the producing region. Olive oil jars were bulbous and wine jars were more columnar. After about 400 A.D., the diversity of shapes decreased and they superseded by cylindrical shapes [1]. During the season of 2008 at El-Bahnasa excavation site, an amphora was discovered and it belongs to the Late Roman period in Egypt (4^{th}-6^{th} centuries AD). The village of El-Bahnasa is situated on the west bank of the Nile, to the west of the road between Maghagha and Beni Mazar in Minia Governorate, Middle Egypt. Amphora was suffered from many deterioration phenomena including, accumulation of dirt and soil residues, narrow and wide cracks, decay and fragility of pottery body, flaking and lamination of the body resulting from the re-crystallization of salts within the pores. Furthermore, many shards are broken from the body and some are missing. The main aim of this study is to identify the chemical and the mineralogical composition of the clay body of amphora, the soil residues and the crystallized salts furthermore, carry out restoration process to it. The chemical, mineralogical and structural characterization of ancient pottery can shed light to the provenance of raw materials for ceramic production and determine the technological processes related to pottery manufacture [5][6]. Different analytical methods were used including; X-ray diffraction (XRD) and scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS) to study different samples of clay body, soil residues and crystallized salts. Complete restoration processes were carried out on amphora, such as cleaning, consolidation of the fragile body, bonding of broken parts and replacement of the missing areas. Paraloid B-72 (an acrylic co-polymer of ethylmethacrylate and methylacrylate (70/30) was used for consolidation and bonding processes and it is used frequently in the consolidation treatment of ancient pottery and ceramics [7][8].

2. Materials and Methods

2.1. *Discovery of amphora*

Through the excavation works which were carried out by the Spanish mission at the El-Bahnasa archaeological site during the season of 2008, an amphora was discovered. The amphora dates back to Late Roman period (4^{th}-6^{th} centuries AD) and it has belly body with a long narrow neck and tapered bottom shape. It has two handles but one only is present and the other was lost completely. The paste has a rough texture and reddish color and it is wheel-made with thick wall. The height of amphora before restoration was about 78 cm. and after restoration is 92 cm., the diameter of the body is 90 cm. while the diameter of the rim is 10 cm. and the height of the neck is 30 cm, fig.

(1-a, b, c. d). The village of El-Bahnasa is situated on the west bank of the Nile, to the west of the road between Maghagha and Beni Mazar in Minia Governorate (Middle Egypt). Today the village occupies part of the archaeological site and it is far around 200 km south to Cairo. The ancient name of El-Bahnasa through the Roman age was Okserynekos, and this name was derived from a holy fish called El-Kanoma which was a god of this place. The Okserynekos was more important towns of Middle Egypt through the Roman age [9][10].

Figure (1) different shapes of amphora and the fragments before restoration processes.

2.2. *Deterioration phenomena of amphora*

Amphora has suffered from many deterioration phenomena including stains and soil residues which were adhered strongly with the surface of the clay body resulting from the burial for a long time in the soil. In addition to cracks, flaking and lamination of body which related to re-crystallization of soluble salts within the pores after rapid excavation in the site, fig. (2-a, b, c, d). The composition and texture reached with the firing temperature is a key factor on salt crystallization decay and hence on the durability of pottery artifacts. Salt crystallization in porous materials is one of the primary causes of stone and ceramic decay, especially in marine environments and that found at archaeological sites [11][12]. Water is vitally involved in most weathering processes. In porous pottery and ceramics, water acts as a transporting agent for chemicals that react with the clay minerals and as a solvent [13]. For example, during burial period, the high level of the ground water can carry the dissolved salts into the pottery pores leaving them (dissolved salt) behind when the saline water evaporates [14]. The soluble salts are hygroscopic and as the relative humidity rises and falls, the salts repeatedly dissolve and crystallize [15]. The newly forming salts eventually occupy a enormous pressures on the fabric of the pottery and these may be sufficient to cause the surface to flake off or to disintegration and fracture of the body [7][16][17]. These salts can be crystallized at just below the surface of the pottery and at times, masses of needle-like crystals can cover the surface, hiding all details which give an unaesthetic appearance to it [11][15][18]. Furthermore, many shards from the clay body of amphora are broken and some are missing, fig. (3-a, b, c)

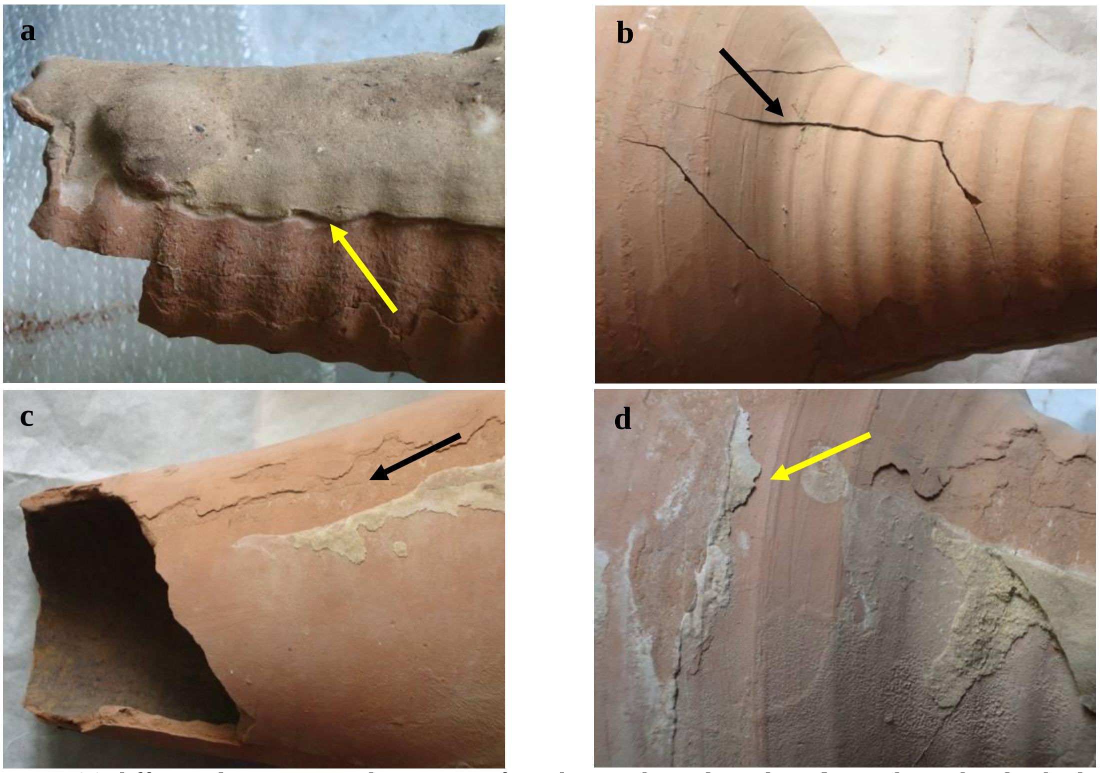

Figure (2) different deterioration phenomena of amphora **a** the soil residues, **b**. cracks in the clay body, **c**. the lamination, **d**. illustrates the crystallized salt on the surface.

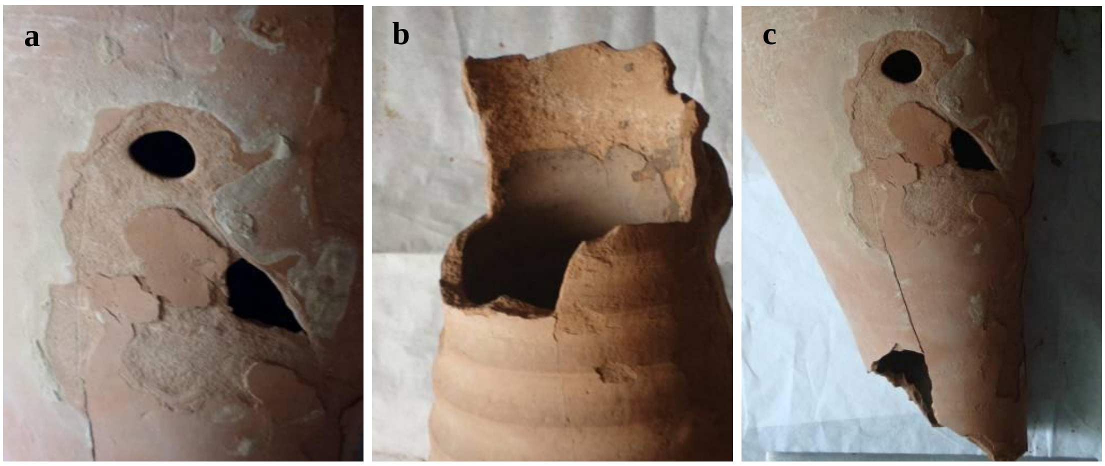

Figure (3) the missing parts of amphora **a** missing parts in the body, **b**. missing parts in the neck, **c**. missing parts in the base.

2.3. *Analytical techniques*

The mineralogical composition of the pottery samples, salts and soil residues were determined by XRD using x-ray powder diffractometer (X' PORT PRO. PANALYTICAL) with Ni-filtered Cu Kα target with secondary monochromator at operating conditions of 45 kV/40 mA. Furthermore, the firing temperature of pottery body was estimated from the mineral phases obtained by X-ray diffraction [19]. Microstructure and chemical constitution of the pottery sample was carried out by scanning electron microscopy (SEM) JEOL, JXA- 840A, Electron Probe Micro Analyzer which coupled with energy dispersive X-ray spectroscopy (EDS) INCA- Sight.

3. Results

3.1. *XRD results*

Mineralogical analysis obtained by X-ray diffraction of ancient ceramic and pottery bodies can provide useful information about raw materials and determine the technological processes related to manufacture [6][20]. The XRD result of sample no. (1) belongs to clay body consists of quartz as a main component, both of calcite and anorthite as minor components as well as halite, magnetite, dolomite and hematite as traces. Decomposition of calcite and dolomite begins at 700 ºC and both phases decomposed completely on firing around 800-850 ºC [21][22][23][24]. This result indicates that the firing temperature of amphora may be around 700-800 ºC. The sample of salt no. (2) contains halite as a major beside quartz and calcite. The sample of soil residues no. (3) consists both of halite and quartz as major components and calcite as trace, all of theses results are listed in tab. (1) & shown in fig. (4).

Table (1) XRD results of body sample, soil residues and crystallized salts

No.	Sample kind	*Identified Minerals*					
		Major		*Minor*		*Traces*	
1	Clay body	Quartz	SiO_2	Calcite	$CaCO_3$	Halite	NaCl
				Anorthite	$CaAl_2Si_2O_8$	Magnetite	Fe_3O_4
						Dolomite	$Ca Mg(CO_3)$
						Hematite	Fe_2O_3
2	Salt	Halite	NaCl	Quartz	SiO_2	Calcite	$CaCO_3$
3	Soil residues	Halite	NaCl	_		Calcite	$CaCO_3$
		Quartz	SiO_2				

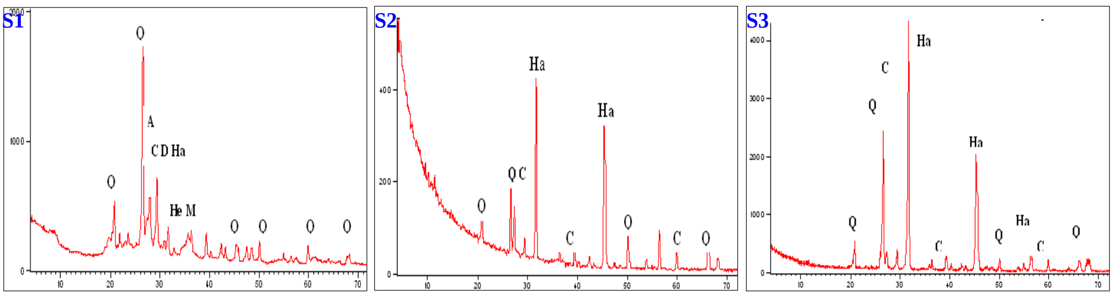

Figure (4) XRD patterns of analyzed samples shows "Q= Quartz, C= Calcite, He= Hematite, Ha= Halite, A= Anorthite, M= Magnetite, D= Dolomite"

3.2. *Scanning electron microscopy results*

The analysis carry out by SEM/EDS provided important information about Microstructure and elemental composition of the ancient pottery and ceramic [19][25]. The sample revealed high proportions of silicon, calcium, aluminum with relatively low concentrations of sodium, potassium, magnesium, chlorine and iron, as shown in fig. (5-a, b, c, d).

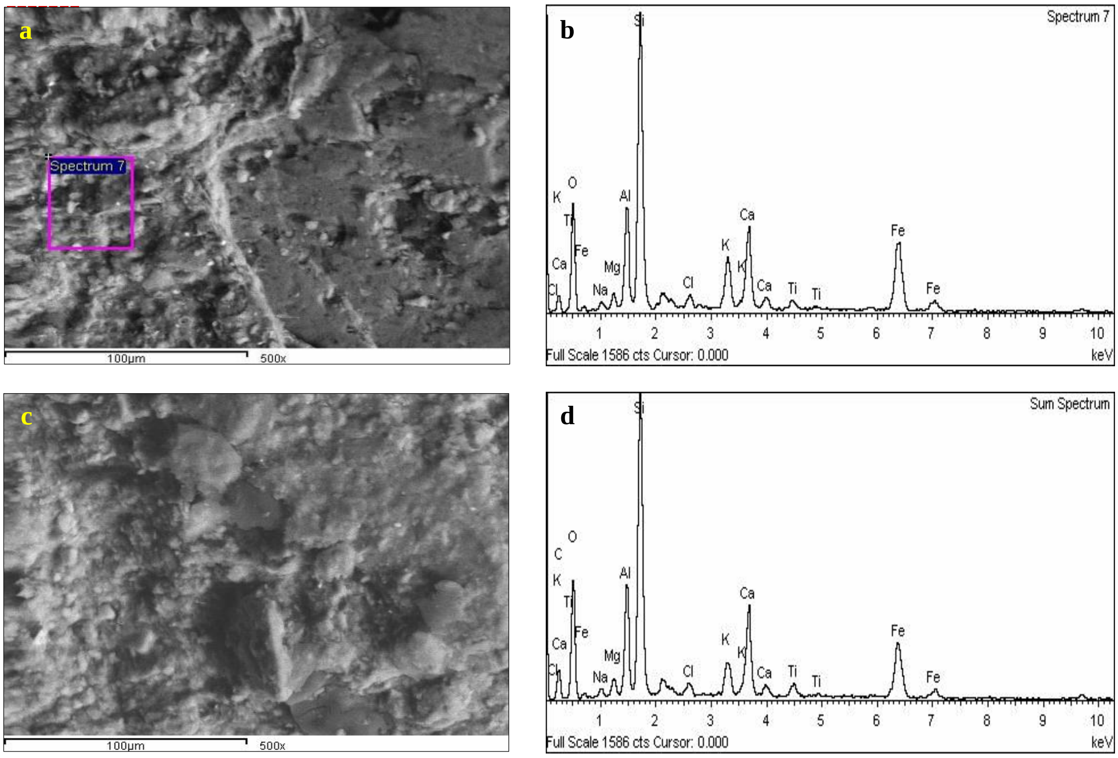

Figure (5) **a**, **c**. SEM images of the red coarse body & **b**, **d**. EDS microanalysis of the body of amphora.

4. Treatment processes

4.1. *Cleaning process*

Before cleaning, it is essential to identify the type of pottery, its mineralogical composition and the nature of dirt and deposits. It is also important to understand the cleaning means that will use to remove the soil residues or deposits and encrustation [7]. So different samples of pottery body, soil residues and crystallized salt were analyzed by XRD. Because of the object was very poor and unstable, initial consolidation with Paraloid B-72 with concentration of 5% was applied before treatment to handle the object. Mechanical cleaning was firstly used to remove the fragile or non- coherent soil deposits on all the body of amphora using brushes with appropriate sizes and dry cotton wool swabs. The advantage of using mechanical cleaning is mainly more easily controlled than chemical methods [26]. The soil residues were deposited on the surface of amphora as a thick layer (around 1- 2 cm) and it was strongly adhered to it. In this case, mechanical cleaning techniques was not enough to remove the previous layer; therefore a wet cleaning method was applied. Cotton poultices immerged in water were effectively used to soften the hard crusts on the surfaces and makes it easy to remove, as shown in fig. (6-a, b). Then, different tools such as a needle, sharp scalpel were required to pick or cut carefully the hard deposits [7]. The thin layers of insoluble surface deposits such as calcium require chemical cleaning methods which involve water, solvents, acids, and alkalis [27]. A useful chemical for removing calcareous deposits is ethylene diaminetetra-acetic acid (EDTA) which is best for removing calcareous material [15][26]. A 5 % solution of the tetra-sodium salts of EDTA which gives a pH of 11.5 was used to remove the black stains and the thin layer of insoluble salts from the surface without seriously affecting the iron content of the object.

Figure (6) illustrates the different treatment processes of amphora. **a**, **b** cleaning of hard deposits with cotton poultices immerged in water, **c** initial consolidation of fragile and lamination body, **d** bonding of the broken shards located at the neck of amphora.

4.2. *Removable of soluble salts*

Identifying the damage caused by soluble salts is easily done visually [28]. The soluble salts (chlorides, phosphates and nitrates) are potentially the most dangerous and they must be removed in order for the pottery to be stable. The method used will depend on the type of object and its condition [7][15]. In this case the soluble salt was halite (sodium chloride). Mechanical cleaning tools such as brushes, needles and scalpels were used to remove crystallized salt and then cotton poultices immerged in water were used to extract the salt through the pores of object.

4.3. *Consolidation*

Consolidation is the process in which the fabric of the ceramic is strengthened by introducing a material into the fabric that will bind together. The most common pottery and ceramics that need consolidation are excavated objects because they tend to lose their binding constituents through leaching or have suffered damage through the absorption of soluble salts [7]. Paraloid B-72 is an acrylic co-polymer of ethylmethacrylate and methylacrylate (70/30) used as an adhesive, consolidant and protective coatings due to their non-wettability, chemical inertness and environmental stability in the conservation of artworks and acetone is the most suitable solvent for it [8][29][30]. Paraloid B-72 was used with concentration of 5% dissolved in acetone for initial consolidation of fragile and lamination body and 10% for fixing chips of the body before treatment as illustrated in fig. (6-c). After all restoration processes were completed, a permanent consolidation treatment was carried out by 5 % Paraloid B-72 in acetone which was applied by the brush.

4.4. *Bonding process*

The broken shards located at the neck of amphora were bonded by using Paraloid B-72 dissolved in acetone with concentration 40% as adhesive. The adhesive was applied to one half of the break only, and the two halves are brought together as shown in fig. (6-d). Paraloid B-72 is suitable for bonding earthenwares objects and the drying time will depend on the ambient temperature, and the thickness and porosity of the pottery [7]. Different narrow and wide cracks on the amphora surface were reinforced by using Paraloid B-72 adhesive where it was applied along the surface of the crack from one side by a fine paintbrush.

4.5. *Replacement process*

Many parts of amphora were lost including the base, neck, rim and body, as well as one of two handles was lost completely. Choosing of the material which will use to fill the missing area depended on the type of ceramic or pottery object that is being treated and the method used to apply the filling material [7]. The missing parts were filled by using polyfilla (calcium sulphate with cellulose fibbers), fig. (7-a, b). The filling materials can be applied to small chips and cracks without using any form of support. Larger fillings often require some forms of support for the filling material until it has cured and this is usually a temporary and external support. Because of the missing parts in amphora were large, unfired clay was used as a support material through curing the filling material used (polyfilla). Before applying the filler, the break edges are dampened first with water to avoid the rapid absorption of water from the filler. Alternatively, the edges were sealed using Paraloid B-72 10 % to makes the filling easier to reverse. Regarding the lost handle, the present handle was used as a guide to can form the new one. The earthenware clay (iron clay) was used and the similar handle was formed by freehand inside the ceramic laboratory at the Faculty of Fine Arts, Minia University. The handle was dried in the open air and then was fired in an electrical kiln at 800 ºC. Finally the handle was polished and fixed in its original place with Paraloid B-72 adhesive, as shown in fig. (7-c). A permanent consolidation treatment was carried out by 5 % Paraloid B-72 in acetone which was applied by the brush, as shown in fig. (7-d). After the restoration process finished, an iron support was made to display the amphora until moving it to the museum and displaying it with appropriate method, as illustrated in fig. (8-a, b, c, d).

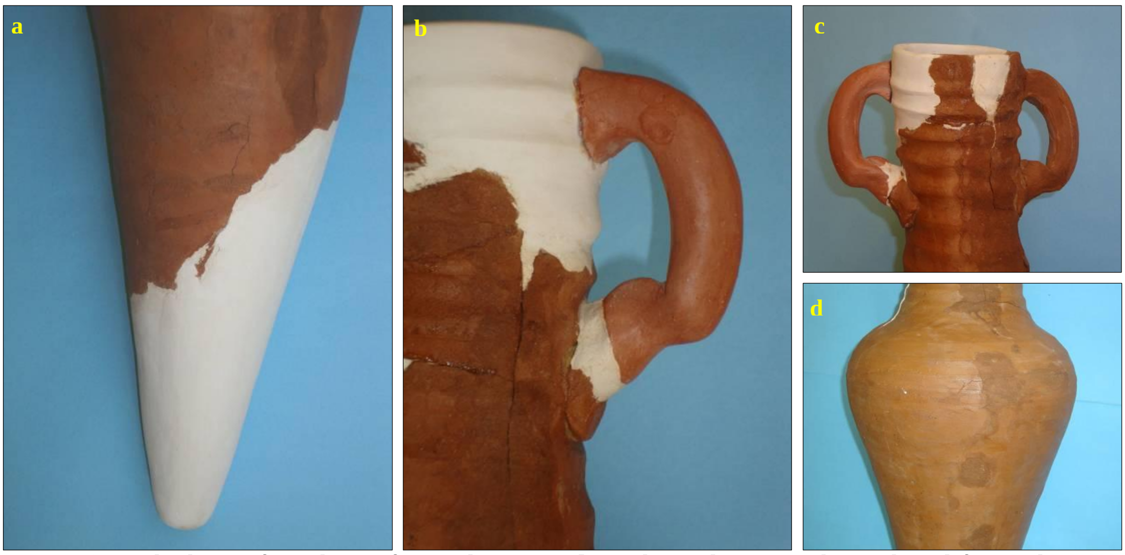

Figure (7) **a** the base of amphora after replacement, **b**, **c**. the replacement the neck and fixing the new handle, **d** a permanent consolidation treatment by Paraloid B-72.

Figure (8) **a**, **b** the amphora after restoration processes, **c**, **d** the displaying the amphora on the iron support

5. Discussion

The obtained results by XRD attested that the local coarse clay rich in iron oxides was used for production of amphora. Furthermore, calcite was used as a temper which was added to the clay through the preparing process. The result obtained by SEM/EDS for body sample showed a good agreement with the XRD result. Because of the object was fragile and unstable, initial consolidation with Paraloid B-72 with concentration of 5% was applied before treatment procedures. Mechanical cleaning was firstly used to remove the fragile or non-coherent soil deposits. Then chemical cleaning by different solvents and aqueous materials such as EDTA were applied to remove the soil residues and stains. The crystallized salt was cleaned firstly by Mechanical tools such as brushes and needles and then cotton poultices immerged in water were used to extract the salt through the pores of the body. Paraloid B- 72 is suitable for bonding earthenwares objects so it was used at concentration 40% dissolved in acetone as adhesive [7]. Polyfilla was used as a filler for the missing parts. It is used in preference to plaster of Paris by many conservators as it is easier to shape, remaining workable for longer and being softer when cured, thus makes it more easily to cut back and carved [31]. A similar handle was formed by using iron clay and it was dried in the open air. It was fired at 800 ºC, and then it was polished and fixed in its original place with Paraloid B-72 adhesive.

6. Conclusion

The amphora belongs to the Late Roman period in Egypt (4th-6th centuries AD) discovered at El- Bahnasa site during season 2008. It was used to ship wine and other products through this period. The results obtained by different analytical techniques (XRD, SEM/EDS) showed that clay used for production of the amphora was rich in iron oxides, whereas calcite was added to the clay as a temper. The firing temperature of the amphora was around 700- 800 ºC. which was estimated from the phases determined by XRD analysis. Amphora was suffered from many deterioration phenomena including stains and soil residues resulting from the burial for a long time in the soil. In addition to cracks, flaking and lamination of body which related to re-crystallization of soluble salts within the pores. Because of the amphora was very poor and instable condition, initial consolidation with Paraloid B-72 dissolved in acetone with concentration of 5% was applied before treatment. Mechanical cleaning tools were firstly used to remove the different soil deposits while chemical cleaning was carried by different solvents and EDTA solution. Mechanical cleaning tools such as brushes, needles and scalpels were used to remove crystallized salt (halite) and then cotton poultices immerged in water were used to extract the salt. The broken shards were bonded by using Paraloid B-72 dissolved in acetone with concentration 40%. A new handle was manufactured by using iron clay and it was fixed by Paraloid B-72 adhesive. Polyfilla (calcium sulphate with cellulose fibbers) was used as a filler to replace the missing parts of amphora. After the restoration processes were completed, an iron support was made to display the amphora until moving it to the museum.

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