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RESTORATION OF TWO TERRACOTTA FRAGMENTS FROM AN AMPHORA FOUND IN A MARINE ENVIRONMENT IN TIPASA

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Abstract:

Due to its favorable geographical location and the significance of the sea in ancient times, Tipasa had several mooring sites along its beaches, bays, and islands. Not far from the beach at the commonly called Sainte Salsa, archaeologists discovered two fragments belonging to the neck of a terracotta amphora of Dressel 1C type, likely used for transporting wine. In this study, it is characterized the terracotta used in the amphora through various investigative techniques: morphology observation under a binocular microscope, petrographic analysis on thin sections, mineralogical analysis using X-ray diffraction, and petrographic analysis (porosity, water absorption, density, etc.). The results indicate that this terracotta was made from clay mixed with tempering agents (sand and straw) to reduce paste plasticity. It was fired in an oxidizing atmosphere at low temperature, as evidenced by the presence of calcite in the shard (incomplete degassing), resulting in high porosity and water absorption. For the restoration of these two fragments, we performed mechanical cleaning of concretions, yielding satisfactory results, followed by a "peeling" mechanical treatment using Klucel®. Subsequently, a complementary chemical treatment was applied to remove remaining $CaCO_3$ carbonates with citric acid and EDTA. While the chemical treatment successfully eliminated dirt and white stains, some areas persisted as white film-like stains. Elemental chemical analysis using SEM-EDS indicated that these areas were primarily composed of silica (SiO_2), suggesting diatoms, organisms with siliceous skeletons challenging to remove with chemical treatment. The use of strong acids was avoided to prevent damage to the calcareous and porous terracotta. Thus, the cleaning process was halted, and the restoration process continued (reassembling, bonding with Paraloid72 at 30%, filling with molding plaster using the balloon method, and finally coloring).

Keywords: *Terracotta amphora; Restoration; Underwater archaeology; Tipasa*

Introduction

In antiquity, the amphora was the most commonly used container for transporting basic goods. It is also referred to as a container. Besides serving as a means of preserving basic products, amphorae had various other uses, such as in masonry works. These can be found in coastal regions, the depths of the sea, and in shipwreck sites. Amphorae are typically made from clay, which is molded, dried, and then fired. Due to their burial in a marine environment for extended periods, they have undergone various forms of damage, including breakage, concretions, stains, and more. The objective of this work is the restoration of two fragments of terracotta amphorae without causing harm to the ceramic shard. The condition of the ceramic at

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the time of its emergence depends on two closely intertwined parameters: the environmental and technological factors. Understanding the object from a technological perspective contributes to a better comprehension of degradation phenomena. The fragility or resistance of an object to external aggressions will be influenced by several factors, including:

- *the composition of the clay*: the presence of naturally occurring or added limestone as a temper in the clay can make it susceptible to acid attacks during chemical treatment. Plant fibers, used as a temper, can also be fragile components of the clay.

- *shaping*: a simple, one-piece form will have higher mechanical strength compared to a more complex form with added elements.

- *nature of decoration*: applied (post-firing) decorations will degrade quickly, while fired decorations will be more resilient.

- *surface finish*: polishing the surface tightens the pores and reduces ceramic permeability to external attacks.

- *firing temperature*: lower firing temperatures result in more fragile objects, and this factor also affects conservation and restoration methods. For example, some treatments and consolidants might be incompatible with ceramics containing calcium, while others could pose a risk to iron, which may be present in the clay, slip, or painted decorations. Consolidants, adhesives, and fill materials must be adapted to the ceramic's fragility level. In the event of stress or impact, their cohesive forces should break rather than those of the ceramic material [1].

Geographic and Historical Context of the City of Tipasa

Located 70 km west of Algiers on the coastal road, as shown in Figure 1, Tipasa occupies a privileged site not far from the Chenoua Massif, which shelters it on the west side. The ancient city extended along the sea for approximately 1500 meters across three promontory hills, separated by deep coves. The city of Tipaza came under the control of the Masaesyli Numidians, where engraved stelae with Libyco-Berber writing were discovered, along with coins from King Massinissa, King Juba II, and his son Ptolemy [2].

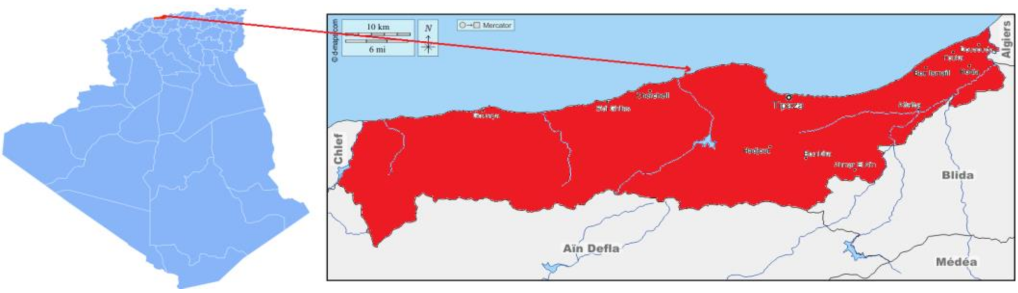

Fig.1. Localisation of Tipasa.

Furthermore, the Mauritanian royal mausoleum is attributed to the reign of King Juba II. Later on, Mauritania Caesariensis became a Roman province after the execution of Ptolemy, and Tipaza thus became a Roman city [3]. It was elevated to the status of a municipality in the year 46 CE and saw significant development, particularly during the reign of Hadrian (117-138 CE) . Tipaza became a colony enjoying Roman citizenship rights between 145 and 150 CE. Christianity appeared in the city of Tipaza in the early 3rd century, and the Church of Saint Salsa bears witness to the persecution faced by converts to this religion during that time. The city was occupied by the Vandals in 430 CE, resulting in the destruction of the city walls [4]. Tipasa came under Roman control, and Catholicism became the official religion during

Hilderic's reign. Later, it was conquered by the Muslims but was used as a quarry by the deys for the construction of the city of Algiers and its surroundings [5]. Currently, the archaeological site of Tipaza is listed as a UNESCO World Heritage site (United Nations Educational, Scientific, and Cultural Organization) [6].

Archaeological Context

The discovery site of the amphora fragments is located in the East Archaeological Park of Tipasa, commonly known as Sainte Salsa. This area primarily functions as an ancient cemetery that extends over a large area [7]. It is situated to the east of the city, bordered by the Mediterranean Sea to the north, National Road 11 to the south, the Islamic cemetery to the east, and the fishing port to the west. The fishing port covers approximately 15 hectares and includes a coastal hill known as Ras Sidi Saeed, also referred to as the "Basilica of Sainte Salsa" district. Terracotta fragments from an amphora was found at the bottom of the sea, at a depth of approximately 5 meters, in the basin formed between the two islets of Sidi Said and the promontory of Sainte Salsa. This basin was once an integral part of the port of the city of Tipasa, and the location of the discovery is indicated in Figures 2 and 3.

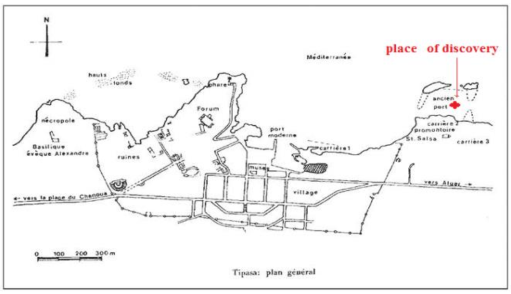

Fig. 2. Place of discovery [8]

Fig.3. Place of discovery by Google Earth [9]

Experimental part

The archaeological ceramic was described after a visual examination.

Visual examination, description, and analyses

The assessment criteria such as dimensions, the description of the paste and its inclusions, as well as the state of preservation are presented in Table1. The visual examination was conducted using a binocular loupe and on a thin section made from a small fragment. Observing the shard under a binocular loupe allowed for a better study of the inclusions present in the paste and an examination of the structural and surface condition of the ceramic shard. Inclusions were described according to the following criteria: color, opacity, shape, size, and frequency. The thin section was analyzed under a polarizing microscope and was able to identify the minerals present in the paste through X-ray diffraction.

Petrographic analysis on thin section

The test was conducted following the standard NF EN 12407: 2000. [10]

Result: under the polarizing microscope, it is observed a high presence of transparent and clear xenomorphic quartz, varying-sized pores, carbonized organic material due to firing, and a matrix composed of aluminosilicate and feldspar (Fig 4).

Table.1. Technical data

Inventory Number: PCT2020
Owner. Laboratory of Historical and Archaeological Studies at Morsli Abdellah University, Tipasa (LEHA)
Designation: Two fragments of Dressel 1C-type Amphora
Paste Color: Greenish-Orange
Function: Likely related to the storage and transportation of wine. Presence of resin inside
Manufacture: Likely wheel-thrown with unrefined clay (variable temper) Oxidative firing, without unglazed decoration, relatively low firing temperature, maximum 800°C

Paste Description (observation under binocular loupe only)
-inclusions white, translucent, sub-angular (250-500 µm), probably quartz
-white to yellowish, opaque, sub-rounded (150-250 µm), probably feldspar
-black, opaque, rounded to sub-rounded (average 400 µm), probably clay aggregates; carbonized black: straw for tempering
-porosity: regular cavities, highly porous (water absorbed directly)
Structural Condition (in fragments): 02 fragments
Deposits (white concretions): covering almost the entire surface and partially the edges, good paste cohesion, erosion of some edges

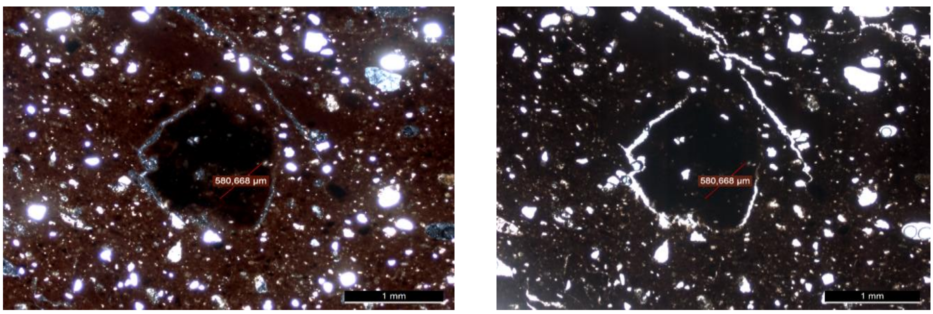

Fig. 4. Shard under LPA on the right and LPNA on the left.

Mineralogical analysis by X-ray diffraction (XRD)

X-ray diffraction was performed on a powder diffractometer, RX D 8 Advance type (Bruker-axs). The results are shown in Table 2.

Table 2. XRD analysis results

mineral	percentage
quartz	+++
kaolinite	+++
illite	+++
albite	++
biotite	+
iron-bearing minerals	+

The Table 2 shows the strong presence of quartz SiO_2, clay minerals like illite and kaolinite, sodium feldspar albite with the formula $NaAlSi_3O_8$, micas, and calcite $CaCO_3$. The presence of calcite confirms that the terracotta shard from the amphora was fired at low temperatures, preventing complete degassing. It's important to be cautious when using acids, especially hydrochloric acid, as it can damage the shard.

Physical characteristics

The petrophysical characteristics are determined according to the NF EN 1936: 1999 [11] and NF EN 13755: 2003 [12] standards, and the results are shown in Table 03.

Table 3. Petrophysical characteristics of the amphora shard

table	bulk density ρ (g/cm3)	specific gravity sp (g/cm3)	water absorption abs %	total porosity pto %
Sample	1,88	2,11	15,23	20,15

It is observed that the amphora shard has low bulk density, high total porosity, and a significant water absorption value. This high water absorption is detrimental when treating the material with chemicals, so it is advisable to work with the material when it is saturated with water

Restoration

In this section, chosen methods for the restoration of the amphora. *Desalination*: salts pose a danger if not removed, risking damage to the object. The methods for extraction haven't always been without risk. In 1984, UNESCO advised, in its Technical Notebook for the Safeguarding of Underwater Cultural Heritage, to prevent ceramics from drying and to wash them carefully. It recommends gradually replacing the immersion medium water with fresh water, reducing the immersion water to zero after two weeks. Then, replace fresh water with distilled water and continue washing until "all" salt has been extracted, as shown in Figure 5.

Fig.5. Desalination of two amphora fragments

Fig.6. Pre-consolidation operation with Paraloid 72

Pre-consolidation: using a 5% solution of Paraloid B72 and a soft, clean brush, apply the solution to the fragile parts of the amphora, as shown in Figure 6.

Mechanical cleaning

Berducou defines cleaning as the operation of getting rid of any degradation product that obscures the object or can be harmful to it, or both [13]. According to Oakley, it involves removing any matter foreign to the object, everything that is not part of the original production [14]. The cleaning process, target initially organic deposits such as algae and moss, which are soft and easy to remove. Was used a descaler for these. For more compact limestone deposits and firmly attached sediments, which are often harder to eliminate, were employed a milling machine (Fig. 7).

Fig.7. Using a milling machine to clean

Mechanical cleaning (peeling)

Klucel® mixed with water forms a water-soluble adhesive that forms a thick gel at very low concentrations. After drying, it forms an adhesive film that cleans the surface of the object. It is then necessary to rinse the substrate with demineralized water to remove any adhesive residue. Result: the test was negative; the adhesive film couldn't remove the white stains. Therefore, were shifted intervention to chemical products.

Chemical cleaning

EDTA Cleaning: Tetrasodium at a concentration of 3.72% (w/v) in a saturated solution was applied on the water-saturated fragment using a compress or cotton swab on a 1 cm^2 surface, were rubbed it thoroughly for 2 minutes, then washed it thoroughly with demineralized water. Result: EDTA cleaning did not yield a positive result, as shown in Figures 8 and 9.

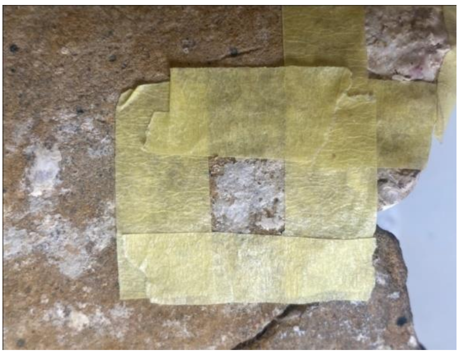

Fig. 8. EDTA cleaning test

Fig. 9. Result of EDTA

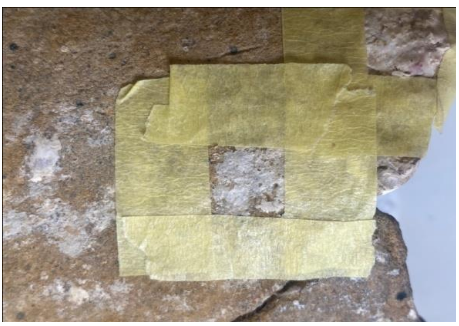

Fig.10. Citric acid cleaning test

Fig.11. Result of citric acid cleaning

Citric Acid Cleaning (10%) did not yield a positive result, as shown in Figures 10 and 11.

Was used hydrochloric acid at a low concentration, HCl 0.01N, applied to the water-saturated fragment with a compress or cotton swab on a 1cm^2 surface, and let it react for a few seconds. Finally, were rinse it thoroughly with demineralized water. Result:a slight effervescence is observed with SEM-EDS. After analysis with SEM-EDS were noted a strong presence of silicon oxide, which gives the idea of the presence of diatoms, very hard siliceous skeletons, which do not react with acid, and very difficult to eliminate (Figure 12).

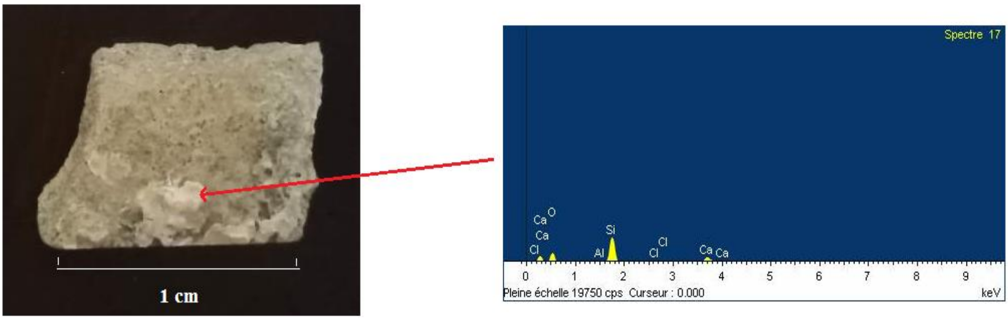

Fig.12. Analysis of the white stains with SEM-EDS.

Table.4. Results of SEM-EDS

Element	**Al**	**Si**	**S**	**Ca**
% weight	0.69	70.54	0.74	25.50
% atomic	0.78	76.86	2.19	19.47

Adhesion Consolidation is a common treatment in the conservation-restoration of archaeological ceramics (especially porous terracottas) that have been weakened by centuries of burial in the ground or, in this case, immersion. For the consolidation of terracotta object, was chosen acrylic glue, which is based on paraloid 72 dissolved in acetone For securely held to ensure the fragments adhere, was used rubber bands to secure them and also was used adhesive tape on the glued surface to hold the fragments in place while the glue dries (Fig. 13).

Fig.13. Fixing the two fragments while the glue dries.

For *filling* the gap on our amphora, were used the balloon method (Figure 14), which acts as a mold. This technique is widely used in the field of ceramic restoration due to its effectiveness. It allows recreating the missing shape using the balloon as a temporary structural support.

Fig. 14. Balloon method

The filling process includes the following steps:

-place a balloon inside the missing area and inflate it to the desired size to support the missing part. Then, was prepared the plaster by mixing it with water until it reaches a slightly thick consistency that can be applied to the balloon without sagging. Carefully apply the plaster using a spatula to recreate the shape and details of the missing part (Fig. 15).

Fig. 15. Plaster application

Let the plaster dry for 24 hours to ensure it dries properly. Gently remove the balloon once the plaster is dry (Figure16), then smooth the surface with sandpaper to achieve a smooth texture and clean the shapes and details of the object Proceed to color the plaster by applying several layers of color.

Fig. 16. The object after restoration

The first layer can consist of a mixture of yellow, brown, and white diluted with a lot of water to achieve a light color

Conclusion

The objective of this work is to restore an ancient amphora using mechanical and chemical methods to remove concretions present on its surface. The results obtained have shown that despite the application of chemicals, some concretions were not removed because they are of a siliceous nature, likely diatoms. Treating them mechanically requires significant force, which can damage the surface as they are well attached to the porosity of the surface. However, through restoration and the use of bonding and gap-filling techniques, it was possible to restore the appearance of the amphora. This study emphasizes the importance of considering different cleaning approaches for fragile historical objects such as amphoras. The results highlight the need to combine mechanical and chemical methods tailored to the specific characteristics of each object, taking into account their state of preservation and composition.

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