PRELIMINARY CONSERVATION TREATMENT AND RECONSTRUCTION OF POTTERY ARTIFACTS EXCAVATED FROM TERRESTRIAL AND MARINE ENVIRONMENTS, LEPTIS, LIBYA

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Abstract

This paper presents a preliminary conservation treatment of four pottery fragments excavated at Leptis, Libya manufactured in varied shapes, sizes, and colours. Studied objects were unearthed as sherds from two different conditions. Determination of the deterioration rates and caused factors of the pottery objects was significant to take the correct decision for conservation treatment. Results demonstrate that marine environment was more deteriorate than sandy soil. This work aims to characterize the chemical structure of pottery objects in the original environment using functional analyses. Mechanical cleaning was the first aid and safe step of conservation procedures. Desalination treatment was used for the long-term stabilization of the excavated pottery; then, internal stresses was decreased. Immersion in distilled water was efficient for decreasing water-soluble salts as halite. Insoluble water-salts were removed mechanically and chemically using dilute hydrochloric acid. Colour and thickness of the potsherds were the clues for the classification for assembling using a PVAc/CN mixture (1:1). Completely reconstruction of the excavated sherds was undertaken for understanding the occupation of the objects, then they were preserved in the storage. After desalination and assembling, painted iron stands were prepared for displaying the treated objects.

Keywords Archaeological pottery; Deterioration mechanism; Re-construction; Desalination treatment; Conservation treatment

Introduction

Pottery finds are one of the most excavated artefacts from marine and terrestrial environments [1], enduring the surrounding conditions. It represents a senior, important, and special type of archaeology science; it is a physical clue to the daily activity in ancient life, and it had an intrinsic role. It can also be a medium for expressing social position or wealth. Pottery has been known since the Neolithic age [2]. In the ancient ages, Libya was famous for commercial and industrial activities. Silphium and olive oil were exported to many neighbouring regions, which required pottery vessels to save them for the long term. Some historians mention that Libyans imported the pottery, but this view cannot be taken because there is much evidence to prove that pottery is mainly locally produced. Many pottery artefacts were found, and the remains of pottery kilns, such as those housed in the archaeological museum of Leptis, contain seals, which confirm that they were made in Libya [3].

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Pottery contains many basic minerals, such as kaolin, montmorillonite, and elite (weathered silicates). Quartz, feldspar, mica, olifin, pyroxene, and some other minerals such as hematite and rutile, calcite, anhydrite, albite (NaAlSi3O8), and apatite (Ca5(PO4)3(F,OH,Cl)) were identified as either course temper added to the pottery or secondary minerals formed by the firing of the clay matrix [4]. Kaolin-serpentine series clay minerals comprise one tetrahedral layer linked to one octahedral layer with no interlayer cations and are termed 1:1layer structures connected by O–H–O bonds. The chemical formula of kaolin is Al2Si2O5(OH)4, whereas the Mg end-member serpentine has the formula Mg3Si2O5(OH)4 [5, 6]. It is the covalent structure of primary building units [7, 8].

The chemical structure of clay varies and induces the colour of pottery to be grey, yellowish, or orange. At the same time, the chemical composition of the clay is not a significant criterion in determining and distinguishing the classification of the pottery type. The raw material contains inclusions that can damage the pot wall during shaping or later firing by cracking. The raw clay is levigated before mixing with water and then kneaded until the mass is smooth [9, 10].

The water that is transported out of the green body of pottery may be affected by the material nature and grain morphology because of stronger interactions of water with particles. These factors could influence the distribution of water and its localization within the body as drying proceeds. Therefore, a similar study on kaolin clay with platelet grains was made. The drying of a pottery green body under constant conditions (temperature, relative humidity, and air velocity) is a process that is undertaken through different stages [11].

A green wave was used to cover the surface to make it smooth. The burning temperature depends on the strength of the interconnects of the child's [12, 13]. The chemical composition of the clay, the additives, and the temperature of combustion often determine the physical properties of the pottery. Fagan stated that the ancient manufacturer added many additives, such as organic matter and some salts [14], as shown in figure 1.

The decorative techniques of pottery involve slip, painting, incision, excision, and scraping. Some pottery tempered with the crushed ashes of tree bark is found [16].

The chemical structure of clay, stages of manufacturing techniques such as drying, the effect of water-dissolved elements and gases, the biological activity of microorganisms, and the burial environment are the main factors contributing to the deterioration of the pottery. Pottery pieces undergo a staining phenomenon because of the chemical structure of clay, firing conditions, additives, and the percentage of iron deposits in the soil of archaeological sites [17]. Both the burial periods in soil and the soaking in seawater affected the physical and mechanical properties of the pottery. But the impact of the marine environment is significant in increasing rates of manifestations of damage such as calcite and sulphide salts, clear vertical cracks, and
erosion in some parts. Soil is an aggregate of organic and inorganic materials, water, and gases. Some soil parts play a positive role in the burials. Pottery is a fairly easy material to break because it is characterised by an earthenware structure that is vulnerable. Pottery is a porous inorganic matter that absorbs saline water through capillary properties, creating internal stresses. Salt efflorescence is related to porosity, chemical structure, and surrounding conditions. Through the burial conditions in the marine environment or salty soil, the adsorbed salt appeared after dehydration [18, 19]. Contaminations such as salts, which are hygroscopic materials, attack porous surfaces [20]. The increasing accumulation of salt affects the drying rate by lowering the vapour pressure of the saline solution, hence reducing the rate of moisture loss [21].

Damage and the deposition of inessential matter are not uncommon by-products of conservation treatment [22]. When dehydration of the object occurs after excavation without desalination treatment, the crystallisation of salts and the movement of saline solutions inside the pores make up on the surface and cause pressure cracks in the interior structure, causing physical damage [23, 24]. To decrease the effect of salts, it is preferable to treat pottery as soon as it is recovered. After extracting the pots from the distilled water and completely extracting the soluble salts, they are left to dry slowly in the laboratory [25].

There was no conservation lab. for archaeological materials in Libya (2013); the conservation facilities were poor. Mechanical cleaning is reliant on the initial state of objects’ damage, the necessity of conservation treatment, the acceptable level of skill and individual performance of conservators and restorers, accuracy, and precision [22]. For instance, if mechanical cleaning techniques were used inexpertly or carelessly, there is a danger of scratching, abrading, removing some of the object surfaces, or physically damaging the fabric of pottery [26].

Dynamic water, tap water, immersion in distilled water, and paper poultices are used to extract water-soluble salts from the pores of the pottery. Chemical treatment is convenient for removing insoluble water salts. Sodium hexametaphosphate (Calgon) is used to dissolve and remove calcareous deposits. Citric acid can effectively desorb organic material being held to a magnesium silicate by outer shell adsorption. However, if rust is the staining culprit, hydrogen peroxide performs well, either used at concentrations (5-10% v/v) for removing the different stains [27].

After finishing the chemical cleaning, a distinction is made between the reconstruction, or mending, and restoration stages. Mending or reconstruction is undertaken to obtain data on vessel form and size and, for purposes of “cross mending,” to establish associational and chronological relationships among strata and features within a site [28].

Many materials were used in assembling pottery, such as bitumen or lead clamps [29]. Also, based adhesives are used to assemble the sherds of broken pottery, like clay, black cement animal glue, Araldite, UHU, cellulose nitrate (CN), Poly vinyl acetate (PVAc), and acrylic such as Paraloid B-72. A PVAc/CN mixture of poly vinyl acetate (PVAc) and some cellulose nitrate (CN) is relatively stable [30]. Gap filling is not essential to complete the conservation procedures when pottery has no problem with the mechanical balance.

This study presents the chemical characterization of the burial environments, soil and seabed, and pottery objects. The bad state of preservation after excavation of various pottery artefacts excavated from different environments and the theme of quantifying the degradation by means of diagnostic-analytical technologies and subsequent treatment were studied.

**Materials and Methods**

Investigation and analyses were used to characterise the chemical structure of pottery and adhering burial sediments to understand the deterioration mechanism. Separated solid samples from the excavation sites and archaeological pottery objects were analysed by the X-
Ray fluorescence (p-XRF) device. Besides, powdered samples were analysed by X-ray diffraction (XRD) to obtain the mineralogical composition of pottery sherds.

**Thermal Test of Reassembling the pottery fragments**

An experiment was conducted to assess the resistance of the PVAc/CN mixture to withstand the high temperature. Modern pottery sherds were assembled by a PVAc/CN mixture in regular reciprocal drops. Then it was placed in the oven at 100°C for 30 minutes, followed by exposure to rapid cooling.

**Optical microscope**

It is considered one of the most important methods of examination, through which it can quantify the texture of pottery, the layers, and the alien vehicles attached to the surfaces of fired clay pots before burning [31, 32].

**X-Ray radiography**

X-ray imaging was used to identify the case of pottery objects in terms of the presence of fine or large cracks or decorations under soil calcification. Sirona X-Ray radiography was used to examine the sherds before the conservation treatment.

The analyst selects a single point within the sherd or vessel, and the software identifies adjacent pixels within a range of gray levels. This process is iteratively repeated with an increasing range of grey levels until the region bleeds into the background, as identified by a very large increase in the area of the region in a single iteration [33].

**X-Ray diffraction**

XRD diffraction analysis permitted the identification of the chemical structure of pottery pots and the diagnosis of archaeological pottery damage. A PHLIPS PW1800 X-ray powder diffractometer was used for the characterization of the chemical structure of pottery. At the beginning of the analysis of the 2θ angle at 2 and the end at 80, the temperature at 20.2°C, the relative humidity at 42%, and the use of copper as a positive electrode for the analysis tube, the sample was prepared by a well-injected and placed in the designated area [34]. Samples from pottery objects, burial soil, and the seabed were analysed by XRD. Specimens of pottery have been crushed in agate mortar to obtain the fin powder [35].

**X-ray fluorescence**

The procedure used to estimate the missing values for the other element in the data set was to replace any missing value by the minimum detection limits (DL) determined by XRF [36]. It is a non-destructive method where the device is used without taking a sample and without transferring the artefact to avoid being damaged.

**TDS device**

This device is suited to measure the proportion of salts, especially sodium chloride, in water or liquid solutions (part of a million). It is divided into two parts: the lower part is intended for water sample placement, and the other is intended for the screen.

**Materials in study**

Excavation was undertaken by the Archaeology and Tourism Faculty at Mergib University from 2012 to 2015. The archaeological site of excavation is located in Leptis city, northwest of Tripolis (coastal area), as shown in figure 2. It represented an important Roman seaport, and it is the most important archaeological site in Libya; it is the largest ancient Greek city [37].

The finds were not reported, and there was no in situ conservation after excavation. The objects were preserved in storage for one year without treatment, so they were housed in a store located at the faculty of Archaeology and tourism at Mergib University. The most excavated objects in Leptis City are inorganic. There is an inflow of such finds in this area, ranging from ruins of temples to glass, pottery, and metals. Among the set of finds, two objects were selected...
showing different aspects of damage, which differ in typology, occupation, and deterioration rates in pottery dating back to the Byzantine period in comparison to the form and decoration.

Beakers are commonly found in the excavation sites of Leptis City. Written and photographic documentation were performed in situ. It consists of three parts: the base, body, and nozzle connected with a semi-circular handle. The object is ornamented with circular grooves that extend from the base to the body. Sherds were laid out according to the colour of the surfaces (firing degree) and their thickness. They were subdivided by sorting the sherds in each group into the rim (from the top of the beaker), base sherds (from the bottom), fragments of handles, and body (sherds that cannot be recognised as the base, rim, or handle). The broken beaker was excavated in 14 pieces, as shown in figures 3 and 4.

In figures 5 and 6, the second object emphasises decorative form; it is a neck-amphora that consists of the body, the nozzle, and vertical mass handles. The marine neck-amphora is free of painted decorations. Disintegration of the excavated material is caused by marine weathering of the object's surface and edge. White and grey stains were seen on the surface.

In figures 7 and 8, a small-scale oil lamp was made of red. It consists of two parts: an upper opening from the top—the first one in the middle to the exit of the wick—and the other opening found at the edge to enter the oil. It was decorated with two different circles in diameter; the outer one represents the perimeter of the oil lamps. There are many orbit points; the distance between them is about 0.4cm. Details of an oil lamp decorated with impressions made with a dentate spatula. The mass handle is parallel with the edge opening. There is a missing part in the middle, the weakest part of the object.
Unknown big sherds were investigated by X-Ray radiography, as shown in figure 9a, b and c illustrate a salt-laden sherd. When the humidity evaporates, the salt efflorescence is seen, indicating the effect of a saline environment.

**Results**

*Optical microscopy (OM)*

Images taken with an optical microscope (OM) show the irregular structure inside the pottery objects. Sherds were investigated by an optical microscope and revealed quantities of quartz in the surface and interior parts. Figure 10a illustrates that fired clay mixed with iron oxides provides a red colour and silica provides a yellow colour. Figure 10b shows the irregular structure and pores. lime, clay minerals, and halite.
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**Fig.10.** Investigation of the selected samples:
a) the interaction of fired clay and quartz; b) irregularly formed inside the pores

**X-ray imaging**

Within the investigated sherds by X-ray radiography, figure 11b indicates that there are no large or fine cracks in the unknown sherds. White grey is well shown where the mass handle is welded to the object. The colour of the image changes with the rotation of the piece; there is a hue of grayscale to reach black. In figure 11d, the black area is caused by the low firing temperature of the potsherd; green straw appears after firing, seen as white areas.

**Fig.11.** X-ray imaging shows checking broken parts from the excavated collection:
a and b show the examination of unknown sherds (photo and X-ray radiography);
c and d show the examination of the unknown sherds (photo and X-ray radiography)

**X-Ray diffraction**

Quartz and calcite were the major minerals in both seabed and soil burial environments, in addition to microcline, albite, dolomite, and halite [38]. Moreover, ZrSiO$_4$ and ZrO$_2$ resulted from the natural zircon sand in the seabed; they differentiated this environment. Both seabed and coastal environments are similar in the same chemical structure, but they differentiate in the identification of titanite, rutile, and polyhalite based on the geographic area. The seabed was indeed composed of white sand with a high percentage of salt. At the same time, halite (2.45%) was detected in the seabed sample; it is an essential component of seawater. X-ray diffraction was used to identify the chemical structure of the original environment, as shown in the following table 1.
Table 1. XRD semiquantative result of samples extracted from the original environment of the pottery (seabed and soil)

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Chemical structure</th>
<th>Seabed</th>
<th>Soil</th>
<th>Seabed</th>
<th>Soil</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>SiO₂</td>
<td>55.21</td>
<td>50.02</td>
<td>39.8</td>
<td></td>
</tr>
<tr>
<td>Calcite</td>
<td>CaCO₃</td>
<td>19.63</td>
<td>29.47</td>
<td>39.8</td>
<td></td>
</tr>
<tr>
<td>Microcline</td>
<td>KalSi₃O₈</td>
<td>-------</td>
<td>9.90</td>
<td>9.95</td>
<td></td>
</tr>
<tr>
<td>Albite</td>
<td>NaAlSi₃O₈</td>
<td>-------</td>
<td>8.04</td>
<td>4.98</td>
<td></td>
</tr>
<tr>
<td>Zircon silicate</td>
<td>ZrSiO₄</td>
<td>9.82</td>
<td>------</td>
<td>-------</td>
<td></td>
</tr>
<tr>
<td>Zircon Oxide</td>
<td>ZrO₂</td>
<td>6.13</td>
<td>------</td>
<td>-------</td>
<td></td>
</tr>
<tr>
<td>Titane</td>
<td>TiCaSiO₅</td>
<td>-------</td>
<td>8.94</td>
<td>-------</td>
<td></td>
</tr>
<tr>
<td>Polyhalite</td>
<td>K₂CaMg(SO₄)₂</td>
<td>-------</td>
<td>6.00</td>
<td>-------</td>
<td></td>
</tr>
<tr>
<td>Rutile</td>
<td>TiO₂</td>
<td>1.23</td>
<td>3.00</td>
<td>-------</td>
<td></td>
</tr>
<tr>
<td>Dolomite</td>
<td>CaMg(CO₃)₂</td>
<td>1.84</td>
<td>2.73</td>
<td>3.98</td>
<td></td>
</tr>
<tr>
<td>Halite</td>
<td>NaCl</td>
<td>2.45</td>
<td>1.49</td>
<td>1.49</td>
<td></td>
</tr>
<tr>
<td>Hematite</td>
<td>Fe₂O₃</td>
<td>3.68</td>
<td>------</td>
<td>-------</td>
<td></td>
</tr>
</tbody>
</table>

As shown in the following table 2, the analysis by X-ray diffraction showed that the clay used in making pottery contains many primary minerals, such as quartz and some other minerals. Quartz and albite are the major minerals in the chemical structure. Besides, calcite and microcline were detected in the beaker minerals. Orthoclase and hematite were detected in the chemical structure of the oil lamp.

Table 2. XRD semiquantative result of powdered samples of beaker and oil lamp

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Chemical structure</th>
<th>Beaker</th>
<th>Oil lamp</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>SiO₂</td>
<td>61.35</td>
<td>44.08</td>
</tr>
<tr>
<td>Hematite</td>
<td>Fe₂O₃</td>
<td>-------</td>
<td>10.62</td>
</tr>
<tr>
<td>Albite</td>
<td>NaAlSi₃O₈</td>
<td>14.72</td>
<td>31.98</td>
</tr>
<tr>
<td>Orthoclase</td>
<td>K₂AlSi₃O₈</td>
<td>-------</td>
<td>13.32</td>
</tr>
<tr>
<td>Calcite</td>
<td>CaCO₃</td>
<td>14.11</td>
<td>-------</td>
</tr>
<tr>
<td>Microcline</td>
<td>KalSi₃O₈</td>
<td>9.82</td>
<td>-------</td>
</tr>
</tbody>
</table>

Albite, quartz, and calcite are the major minerals in the chemical structure of the neck-amphora’s body. White stains are composed of aragonite and dolomite, calcite, and quartz. Iron oxides are grown on surfaces composed of lepidocrocite, goethite, and albite associated with calcite, as illustrated in table 3.

Table 3. XRD semiquantative result of powdered samples of marine neck-amphora

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Chemical structure</th>
<th>Body</th>
<th>White stains</th>
<th>Brown stains</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>SiO₂</td>
<td>35.30</td>
<td>3.91</td>
<td>7.25</td>
</tr>
<tr>
<td>Dolomite</td>
<td>(Ca,Mg)CO₃</td>
<td>------</td>
<td>22.35</td>
<td>7.25</td>
</tr>
<tr>
<td>Calcite</td>
<td>CaCO₃</td>
<td>25.24</td>
<td>8.94</td>
<td>15.39</td>
</tr>
<tr>
<td>Aragonite</td>
<td>CaCO₃</td>
<td>------</td>
<td>64.80</td>
<td>15.39</td>
</tr>
<tr>
<td>Lepidocrocite</td>
<td>FeO(OH)</td>
<td>------</td>
<td>57.97</td>
<td>15.39</td>
</tr>
<tr>
<td>Goethite</td>
<td>FeO(OH)</td>
<td>------</td>
<td>------------</td>
<td>15.39</td>
</tr>
<tr>
<td>Albite</td>
<td>NaAlSi₃O₈</td>
<td>39.46</td>
<td>19.39</td>
<td>19.39</td>
</tr>
</tbody>
</table>

Chemical constituents of the extracted pottery are categorised as present in major (quartz, calcite, and clay deposits), the same composition of the find site as illustrated in figure 13.
As shown in table 4, elemental concentration analyses were performed by X-ray fluorescence, and the results obtained confirm XRD results. The chemical structure of pottery objects is different. The presence of gold with amalgam proves that the beaker was likely used in the extraction of gold. In addition to As, Co, Th, Rb and Nb being detected, they were likely associated with gold ores.

**Table 4.** Results of X-ray florescence for samples from the archaeological pottery objects

<table>
<thead>
<tr>
<th>Elements</th>
<th>Beaker, ppm</th>
<th>Oil lamp, ppm</th>
<th>Candlestick, ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mn</td>
<td>384</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>Zr</td>
<td>372</td>
<td>28.6</td>
<td>48</td>
</tr>
<tr>
<td>Sr</td>
<td>153</td>
<td>17.5</td>
<td>39</td>
</tr>
<tr>
<td>Al</td>
<td>96.05</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>Zn</td>
<td>93</td>
<td>54</td>
<td>87</td>
</tr>
<tr>
<td>Si</td>
<td>127</td>
<td>170</td>
<td>151</td>
</tr>
<tr>
<td>Pb</td>
<td>-----</td>
<td>28</td>
<td>-----</td>
</tr>
<tr>
<td>As</td>
<td>63</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>Co</td>
<td>51</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>Au</td>
<td>48</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>Th</td>
<td>42.43</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>Rb</td>
<td>35</td>
<td>26</td>
<td>30</td>
</tr>
<tr>
<td>Nb</td>
<td>10</td>
<td>12</td>
<td>-----</td>
</tr>
<tr>
<td>Hg</td>
<td>21</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>Mo</td>
<td>-----</td>
<td>4</td>
<td>-----</td>
</tr>
<tr>
<td>Fe</td>
<td>3.57</td>
<td>2.93</td>
<td>3.44</td>
</tr>
<tr>
<td>Ti</td>
<td>0.3</td>
<td>0.28</td>
<td>-----</td>
</tr>
</tbody>
</table>

**Conservation treatment of the pottery**

Sherds of broken pottery likely necessitate direct conservation to avoid loss from continued fragmentation or abrasion of edges [39].
Removing the interior soil encrustations

Conservation treatment was not performed after excavation in situ. Since there were calcifications of encrustation on the exterior and interior of the pottery, mechanical treatment was the first aid to conservation. Hand tools have been used, such as scalpels, fibreglass, and toothbrushes, to remove calcifications of the earthen materials and salts from the inner of the beaker and oil lamp without loss of the pottery, as shown in figure 13. Also, 3% hydrogen peroxide and soap were convenient to remove the inside encrustations completely. Soot and burnt surfaces were treated using 2% acetone.

Fig. 13. The stages of mechanical cleaning of pottery objects: 
a and b - integral state, clay encrustations inside the beaker; c - integral state of the oil lamp; 
d, e, and f - removal of unwanted soil encrustations inside the oil lamp respectively

Desalination treatment by immersion in distilled water

Pottery is a porosity material that leads to good penetration. Objects extracted from the burial environment were saturated with dissolved salts. When pottery was exposed to the air environment, the danger of salts appeared to be recrystallization above the surface and inside the pores. The most important salts that dissolve in water are chlorides, nitrates, and phosphates. Water-soluble salts were removed by immersion in distilled water. Then, distilled water was changed to renew the activation of salt removal, as illustrated in figure 14.

Monitoring the desalination progress was undertaken by TDS. Firstly, the salt ratio was lower, but it increased over time. The distribution of salts remaining after treatment is shown in table 5.

<table>
<thead>
<tr>
<th>Duration</th>
<th>Ratio of salt (ppm)</th>
<th>Beaker</th>
<th>Oil lamp</th>
<th>Neck-amphora</th>
</tr>
</thead>
<tbody>
<tr>
<td>At the beginning immersion</td>
<td></td>
<td>83</td>
<td>93</td>
<td>177</td>
</tr>
<tr>
<td>24 hrs</td>
<td></td>
<td>177</td>
<td>257</td>
<td>629</td>
</tr>
<tr>
<td>48 hrs</td>
<td></td>
<td>75</td>
<td>136</td>
<td>472</td>
</tr>
<tr>
<td>72 hrs</td>
<td></td>
<td>46</td>
<td>94</td>
<td>123</td>
</tr>
<tr>
<td>96 hrs</td>
<td></td>
<td>23</td>
<td>26</td>
<td>36</td>
</tr>
</tbody>
</table>
The removal of unwanted encrustations of insoluble salts was performed by chemical treatment. Tap water was not efficient for completely removing the desalination treatment; the ratio of salinity was higher (164ppm).

![Fig. 14. Desalination procedures of the beaker, oil lamp, and neck-amphora](image1)

![Fig. 15. Calcification of salts inside the neck-amphora (a), after treatment using dilute hydrochloric acid (b)](image2)

**Removing soot and water-insoluble salts**

Firstly, organic solvents such as xylene and acetone were used to degrade the appearance by removing charred and earthen deposits from the beaker surface. Without pre-soaking in water, insoluble-water salts like calcium carbonate encrustations were not completely removed. Then, they were mechanically cleaned followed by chemical treatment using dilute hydrochloric acid (2%). Finally, the sherds were immersed in distilled water to remove the chemical residues.

**Desalination treatment of marine neck-amphora**

The desalination treatment of the marine neck-amphora is different from that of the terrestrial pottery. Salts dissolved in water move to the porous pottery due to the capillary property and then crystallise in the pores during the drying process [39].
The object was immersed in 50:50 seawater/distilled water for three days. Then, it was transferred to pure distilled water, adding biocide to the solution until salts were decreased [40]. Extracting the insoluble salts, such as calcium carbonate and iron stains, was done by dilute hydrochloric acid. After extracting the pottery from distilled water, solvents and a warm air stream were used to dry the pot-sherds shown in figure 16.

![Warm air stream through dehydration of the beaker](image1.png)

**Fig. 16.** Warm air stream through dehydration of the beaker

*Reconstruction of the archaeological pottery*

Reconstruction of the beaker and oil lamp was carefully performed by gluing. To ensure that the adhesive material is suitable for mending the pottery fractures, the use of composite resin depends on the thickness of the wall sherds, which are not thick. Some parts are missing; conservation treatment will be completed after finishing the excavation. They were joined using a PVAc/CN mixture, then the assembly points were hidden.

The oil lamp was broken into three parts; two parts from the base were joined to build the upper part. There is still a missing part that extends from the base to the top.

Firstly, the edges of potsherds were cleaned with acetone. The adhesive was placed with mutual points on the two edges after cleaning, and the two parts were combined well to be affixed. After the adhesive was hardened, the excess was removed using a scalpel or cotton dampened with an alcohol solution, as shown in figure 17. White and blackish stains were not removed completely; it was difficult and needed the use of concentrated chemicals.

![Pottery objects after finishing the conservation treatment](image2.png)

**Fig. 17.** Pottery objects after finishing the conservation treatment
PRELIMINARY CONSERVATION TREATMENT AND RECONSTRUCTION OF POTTERY ARTIFACTS

Fig. 18. Painted iron stand for displaying the treated objects

Long-term Preservation of treated objects

The future conservation of pottery artefacts found in terrestrial and marine archaeological sites appears to be a straightforward material science problem involving elucidation of the structure to develop conservation procedures that prevent deterioration. It is constrained by ethics, aesthetics, and cultural contexts that may complicate, constrain, and ultimately direct preservation strategies [41].

After assembly, pottery objects were preserved without gap filling. They were preserved dynamically to save their aesthetic values. To avoid unnecessary handling, painted iron stands were prepared. The height of the stand of the beaker is 15cm, and the height of the stand of the lamp is 10cm. Iron stands were painted with a suitable coating of black paint to protect the pottery from the effect of iron stains, as shown in figure 19. Also, it would have been preferable to prevent the friction between the treated objects.

Discussions

Clay minerals are the major structure of pottery objects [42]. The chemical composition of clay is not a reliable standard for identifying and distinguishing the type of clay after firing. Clay with a similar chemical composition differs in some of its physiochemical properties as a surface colour according to its preparation and the additives. The excavation site at Leptis City revealed a group of pottery artefacts leached from the marine and coastal environments.

Pottery objects were exposed to different factors and aspects of deterioration that affect the internal structure and physiochemical properties [43]. Of the pottery collection preserved at the storage of the Faculty of Tourism and Archaeology, Mergib University, two objects were selected for this study.

The occupation and nature of ancient pottery made it easier to crack or break. The influence of pH in seawater ranges (4.5–8.5) is imitated by salt-induced degradation of surfaces. As the pieces of the pottery looked to be disturbed, the mutilation was undertaken before the burial; there were missing parts.

After investigation with the naked eye, magnifying lenses, an optical microscope, X-ray radiography, and analysis techniques, it was concluded that the archaeological pottery in its original environments was exposed to various deterioration factors. Calcium carbonate adhered to the surfaces, making it the most popular salt that did not dissolve in water. When self-drying the pottery immediately after extraction from the marine or coastal environment without
treatment, salts crystallise inside the porous, increasing the initial pressure cracks. These phenomena caused by the lack of immediate treatment contributed to an increase in the damage rate.

The marine pottery was leached as one piece; on the contrary, other objects were excavated from the terrestrial environment as small pieces resulting from the pressure of soil layers. Sand in the coastal environment is in a more preservative condition than in the seawater; the continuous movement of the seawater is the main deterioration factor, as is the alteration of the surrounding environment. The seabed of the marine environment contains a high content of quartz, calcite, and halite; the deterioration rate in the marine environment was heavier than in burial. Therefore, marine pottery needed special care in removing salts. To remove water-soluble salts such as chlorides, they were easily soluble and removed by washing and immersion in distilled water [44].

X-ray fluorescence results demonstrate that gold and mercury were detected inside the beaker, and at the same time, the beaker’s surface was covered with soot. These estimates assume that it was likely used in the cupellation of gold.

XRD results revealed that halite was identified in the seabed at 2.45%; this ratio is lower than the known percentage in seawater (3.5%). Most excavated pottery from the terrestrial and marine environments was saturated with salts such as sodium chloride and calcium carbonate [45]. Despite the proximity of the excavation site to the sea, objects were extracted from a dry environment, and their condition is coherent. Salt crystallisation was seen on some unknown potsherds, which led to the physio-chemical properties loosening.

Mechanical treatment was used to remove the accumulation of earthen sediments and reveal the pottery surface. Brushing the surface with tooth and fibreglass brushes was undertaken to remove the adhering foreign materials. Chemical treatment was undertaken using hydrogen peroxide (3%) for removing the calcification; it is an oxidising agent. It was preferable to remove insoluble salts using mechanical cleaning, but chemical treatment with dilute hydrochloric acid (2%) was more efficient. After that, they were immersed in distilled water to remove the residual acid [46].

After visual inspection, we experimented with potsherds that were assembled using a PVAc/CN mixture resisting thermal ageing to ensure that the adhesive material is suitable for use in bonding pottery fractures when a popular polymer such as paraloid B-72 is not found. The surface colour and the wall thickness were the guides for the reconstruction of the sherds. For good joining, potsherds were cleaned carefully before reconstruction. Also, stains on the surface after joining the broken parts of the PVAc/CN mixture as an adhesive were not seen. There was no disintegration of the assembled parts. Further excavation in Leptis City will be conducted to complete the missing parts of the treated objects.

Conclusion

Some pottery artefacts came from the excavation at Leptis City; the geographical location was suitable for the preservation of the objects in the long term. The study demonstrates that both the burial periods in soil and seawater affected the physical and mechanical properties of the pottery, and the impact of the marine environment is significant in increasing rates of manifestations of damage. The main deterioration factors upon excavation were the pressure of the soil layers, which broke the pottery objects. Sandy soil was dry, and the objects were buried in greater depth, far from the effects of the ground water, humidity, and rains. The crystallisation of insoluble salts on the surfaces of neck-amphora in the form of calcifications was the most difficult to remove. XRD results revealed that the chemical composition of the deposits inside the pots matched the chemical structure of the surrounding environment.
The comparative analysis indicates the chemical structure of the pottery and the original environments (burial and seabed). X-ray fluorescence results demonstrate that silicon, zircon, and zinc are the main elements in the chemical structure of the pottery objects. Zircon increases the pottery resistance of the surrounding environment. The surface colour and chemical structure of the objects confirm that the clay likely made from the vessels was not taken from the same source. It also showed that the clay contains a high percentage of sand as well as calcite. Calcium carbonate and iron stains were the most calcified salts found in the marine neck-amphora.

Results demonstrate that the marine environment was more deteriorated than sandy soil due to continuous changes in the surrounding conditions induced by kinetic waves, such as the chemical structure of the seabed, temperature, and the effect of insoluble-water salts such as calcium carbonate and sulphides. The saturation of the marine pottery with salts resulted in a change in the surface colour and increased the specific weight. Mechanical treatment and repeated immersion in distilled water were efficient for the removal of soluble water salts and earthen encrustations.

Organic solvents were used to remove the soot from the beaker, restoring the original colour of the surface. Partial treatment of the pottery; gaps were not filled to dictate finding the missing parts until finishing the excavation. A PVAc/CN mixture was used in assembling the sherds; it’s easy to use and resistant to the surrounding conditions.

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