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Original article

CHARACTERIZATION AND CONSERVATION STUDY OF ANCIENT EGYPTIAN BRONZE BELLS

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Abstract:

Two Bronze Bells were excavated through the Egyptian- Italian expedition in Medinet Madi, Fayoum, Egypt. They are dated back to the Late Period (664-332 BC) preserved in the store museum of Kom Oshim, Fayoum governorate. They were manufactured by the lost wax process. Visual examination, USB digital microscope, metallographic examination, scanning electron microscope with energy dispersive spectroscopy, portable X-ray fluorescence and X-ray diffraction were used in this study to elucidate the micro-structure and elemental chemical composition of the bells, and identify the corrosion morphology. The results indicated that the bells had been manufactured with a ternary alloy Cu-Sn-Pb. Two-phased microstructure the α-Cu phase and the (α-Cu+δ-Sn) eutectoid could be observed with Pb small globules and scattered porosity. The corrosion products were identified as atacamite, paratacamite and malachite for the bronze bell, and goethite for the iron clapper. Conservation treatments included cleaning and protection processes to reveal the bells shape and provide better protection against corrosion.

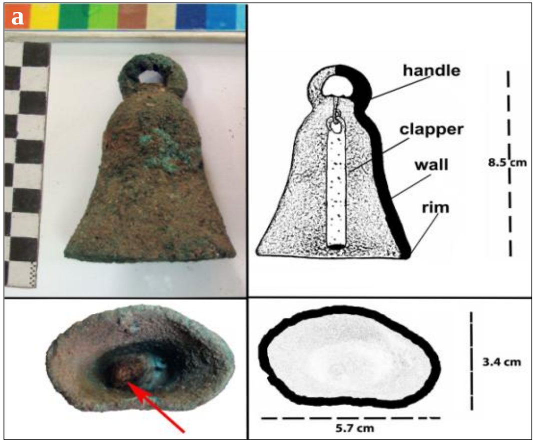
1. Introduction

Bells have been made for about 4000 years. They were usually used in Assyria, Persia, Babylon, China, Greece and Egypt [1-3]. In ancient Egypt, bells were known since the pre-dynastic (before 3100 BC) until the Coptic period (395-642 CE); they have a religious and functional significance. Bells were used not only as a musical instrument but also as a pendant hanging around the neck of the animals of the household and as sacrificial animals like dogs and cows [4-7]. Bronze bells are the most common type of bell but were also occasionally made of gold or silver by emphasized their appearance as small ornaments on Egyptian necklaces [4,5]. Bronzes with varying tin content were produced for different purposes – tools, chandeliers, statues, coins, and later canons and bells. In ancient times, daily used objects such as bells were manufactured from high-tin bronze [8,9]. They are usually compounded of copper and tin, in the proportions of three of copper to one of tin [2]. The addition of Sn leads to further strengthening, preventing deformation of the bell by the clapper which could, in turn, alter the tone. Though, a moderate use of Sn is recommended to avoid fracture tendency during striking. Concerning sound, increasing the tin content leads to an increase in the duration of the sound. However, other elements such as lead, that was commonly found in copper alloys, can be used to increase the fluidity of the molten metal, and thus facilitated casting without detracting from the strength of the alloy. In bell manufacturing, lead would lead to a decrease in the sound duration [10-13]. Traditionally, bell materials consisted of a wide variety of additional alloying elements such as Zn, Bi, Ag, Sb, As, Ni, Fe, P, S and Si. Some of these elements (e.g. Zn, Ni, Fe, Ag, Sb) were introduced on purposes, while others such as sulfur and phosphorus were contaminations introduced accidentally during the bronze melting process, mainly from charcoal and coke [14, 15]. These elements will influence the final quality of bell materials and their effects on the mechanical properties including failure and fracture of tin bronzes [8]. Bells came throughout the ancient world in different shapes with various forms of handles and clappers. The spherical, conical, and various domed forms are some of the bronze bells. An internal clapper, an external hammer, or an interior pellet or pea (for spherical cloves) might give the sound. A bell is more musical than a rattle, even if the two are connected [3, 16,17]. Most ancient Egyptian bronze bells were cast using the lost-wax process, the usual technique for ancient bronze bells and most bronze objects in general [3]. We have a large number of ancient Egyptian bell molds (now in the Cairo museum, cat. #32315 a, b) provides good evidence of metal foundry in ancient Egypt [4]. The quality of bronze casting is closely dependent on the individual steps in the technological process. Sand casting is the widely practiced technique in bells manufacturing, involves five key operations: pattern-making, core-making, molding, melting and pouring [1,2]. The present paper aims to make a characterization and conservation study of two bronze bells by using various laboratory methods to determine metallic composition, microstructure, and corrosion layers; providing useful information for the decision making to select appropriate treatment methods of the bell.

2. Materials and Methods

2.1. *Description*

The two bells under study were preserved in the store museum of Kom Oshim, Fayoum governorate, with No. (89). They were used in daily life purposes, as they were hung around the animals' neck like cows and sheep to give a sound of movement. Such practical uses make it more of a noisemaker than a musical instrument. The first bell, fig. (1-a) measures 8.5 cm length, the base is 5.7×3.4 cm, and weighs 102.59 g. The second bell, fig. (1-b) measures 7.5 length, the base is 4.5×3.7 cm, and weighs 82.59 g. The wall thickness is approx. 2 mm. There are no decorations on the surface. The two bells have the same shape, an open base and cone-shaped without feet, and a loop handle. It is clear from figure (1-a) that there is a deformation in shape (especially in the mouth) in the large bell, which may result from soil pressures in the burial environment. The loop handle, which is the most common handle type, is usually cast in one piece with the bell's body. The big bell has an iron clapper (responsible for making the tone) strung from the interior through a piece of metal hooked throughout a perforation [18,19] as shown in figure (1-a), but it is missed in the small bell as shown in figure (1-b).



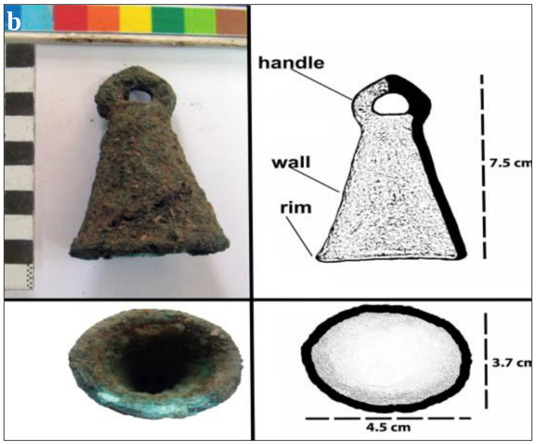


Figure (1) Shows **a.** left: the big bell covered with a thick corrosion crust from interior and exterior, arrow refers to the iron clapper which looks extremely corroded, right: schematic description shows the bell' main parts that consist of a handle, body, rim (base) and clapper, **b.** left: the small bell covered with a thick corrosion crust from interior and exterior, right: Schematic description shows the bell' main parts that consist of a handle, body, and rim (base), but the iron clapper is missed.

2.2. *Examination and analysis*

On-site observations and examinations were carried by using USB digital microscope to identify deterioration conditions. Elemental analysis of the bell manufacturing alloy was undertaken in situ by portable X-ray fluorescence (pXRF) using Bruker S1 TITAN and TRACER 5i Handheld XRF Spectrometer. Analysis has been directly carried out on six different spots on the bell's corroded surface without taking sample. A small, detached sample from the bell base was used for the examination by metallographic and scanning electron microscope. It was prepared as a cross-section to study the metallic microstructure by Leco L31 metallographic microscope. The cross-section was prepared by embedding the sample in epoxy resin and polished using 600, 800, 1200 and 2000 grit emery papers, and polished with 0.5 μm Alumina suspension. The metallographic sample was observed before and after etching. The etching was made using an alcoholic ferric chloride solution (240 ml ethanol, 60 ml hydrochloric acid and 20 g ferric chloride) for 5 sec. SEM observations were made through the Inspect S50 SEM (FEI company). The same pre-prepared sample for metalographic examination and another one from the iron clapper were examined under SEM/EDS. X-ray diffraction (XRD) was undertaken with D8 advance X-ray diffractometer X. (Bruker, Germany). Two samples were analyzed by XRD to identify the composition of crystalline corrosion products of the bronze bell and the iron clapper. Conservation treatments were accomplished to remove soil deposits and corrosion products before protective coatings can be applied to the bells. Such deposits can be removed either by chemical or mechanical techniques. Protection system must be applied to passivate the bells and protect it from further corrosion.

3. Results

3.1. *Microscopic observations*

USB microscopic investigations of the two bells are given in fig. (2-a, b, c). They were completely covered with a thick crust of green corrosion products incorporated with soil inclusions. The iron clapper is extremely corroded and stuck firmly to the bell wall. After simple cleaning, a very small perforation was detected in the small bell's shoulders, as shown in fig. (2-c). It may be attributed to localized corrosion in soil or a result of casting defects.

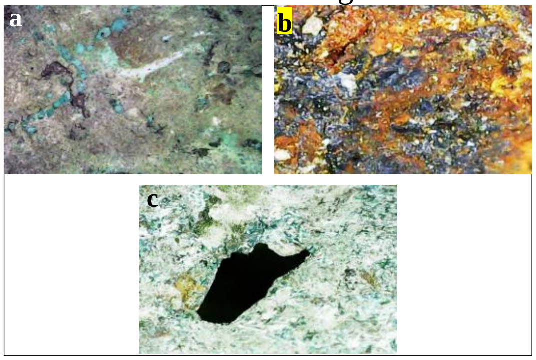


Figure (2) Shows optical microscopic images of the corroded bell surface; **a.** thick corrosion products mixed with soil deposits, **b.** oxide and hydroxide corrosion products of the iron clapper, **c.** very small perforation was observed in the small bell's shoulders.

3.2. *Microstructure of the bell*

Metallographic and SEM/EDS investigations of the bell microstructure are shown in fig. (3-a, b, c) respectively. Energy dispersive spectroscopy (EDS) elemental mapping images showing the distribution of Cu, Sn and Pb concentration in the tin-bronze alloy are given in fig. (4-a, b, c, d).

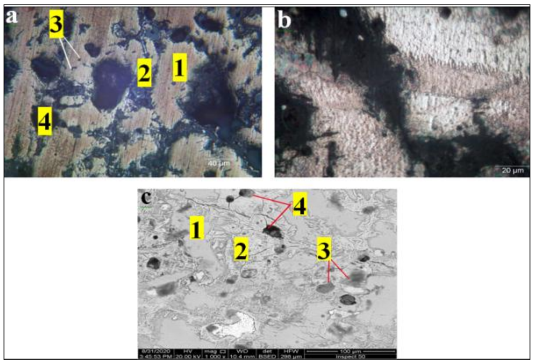


Figure (3) Shows metallographic image of polished but un-etched sample; **a.** **1)** α-Cu phase, **2)** α-Cu^{+} δ-Sn phase (eutectoid), **3)** Pb globules (light dark), **4)** Porosity (dark), **b.** metallographic image of polished and etched sample at a higher magnification, **c.** SEM image confirm the same above microstructure

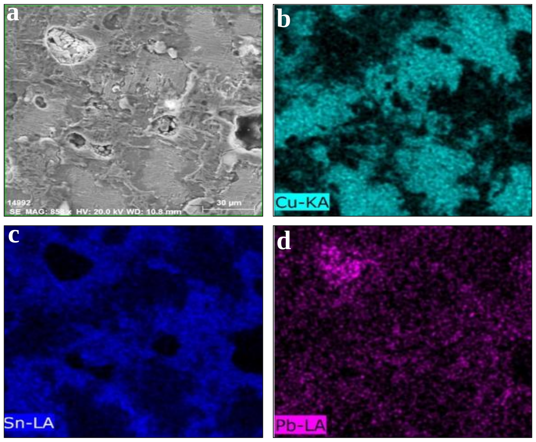


Figure (4) Shows EDS elemental mapping images of the distribution of chemical elements in the alloy metallic core

3.3. *Chemical compositions*

Elemental analysis by *p*XRF on the corroded surface will recognize the metallic elements attributed to the bulk alloy, corrosion products and soil deposits, tab. (1-a). It is clear that the main alloying elements of bell material were Cu, Sn and Pb. The Cu content varies from 66.81 wt. % to 81.53 wt. %, the Sn content varies from 5.49 wt. % to 13.66 wt.% and the Pb content varies from 1.36 wt. % to 3.76 wt. %. The high silicon and calcium content vary from 2.67 wt. % to 5.30 wt. % and from 2.18 to 4.19 wt. % respectively. EDS analysis results of the metallic core are given in fig. (5) and tab. (1-b). It can be noted that the bell main elements are copper, tin and lead, where Cu is 61.71 wt. %, Sn is 29.31 wt. % and Pb is 8.98 wt. %. Iron and nickel are found as impurities. EDS analysis of the iron clapper are presented in fig. (6) and tab. (1-c). The iron content varies from 48.48 to 92.98 wt. %.

Table (1-a) *p*XRF results of different spots on the two bell's corroded surface (wt %).

Spot (*Element wt %*)	**1**	**2**	**3**	**4**	**5**	**6**
Mg	0.568	0.089	0.295	0.971	0.117	0.420
Al	0.414	0.809	0.677	0.332	0.789	0.179
Si	5.302	4.453	4.360	2.672	3.311	3.110
P	0.191	0.122	0.173	---	0.044	0.140
S	0.432	0.364	0.143	---	---	0.883
Cl	5.531	7.429	6.652	3.990	3.072	8.452
K	0.699	0.644	0.606	0.178	0.338	0.497
Ca	3.071	2.178	3.170	2.449	4.190	2.814
Fe	0.638	0.592	0.698	0.187	0.430	0.474
Ni	0.252	0.126	0.265	0.209	0.117	0.416
Cu	66.813	69.704	68.492	81.526	70.158	72.064
Sn	13.344	11.497	13.111	5.487	13.658	9.076
Pb	2.743	1.981	1.356	1.987	3.764	1.473

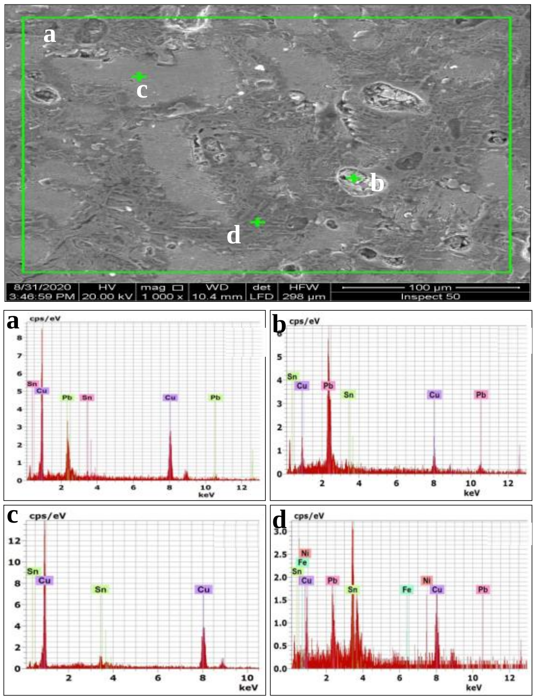


Figure (5) Shows SEM image the corresponding EDS analysis of some phases in the bell's metallic core.

Table (1-b) EDS analysis results of the chemical composition of the bell.

Spot *(Element wt %)*	Fe	Ni	Cu	Sn	Pb
A	---	---	61.71	29.31	8.98
B	---	---	23.08	6.75	70.17
C	---	---	87.75	12.25	----
D	0.89	0.22	40.29	46.72	11.88

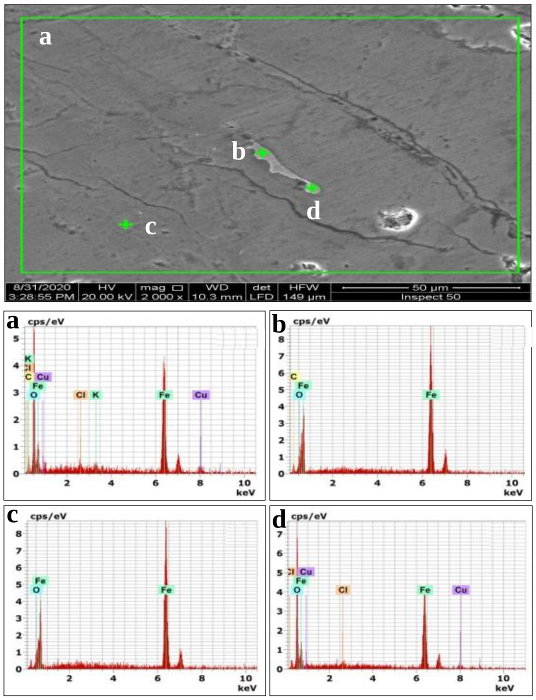


Figure (6) Shows SEM image the corresponding EDS analysis of different spots in the iron clapper.

Table (1-c) EDS analysis results of different spots in the iron clapper.

Spot *(Element wt %)*	C	O	Cl	K	Fe	Cu
A	10.60	36.00	0.70	0.64	48.48	3.58
B	10.55	7.52	---	---	81.94	---
C	---	7.02	---	---	92.98	---
D	---	42.03	0.87	---	54.41	2.69

3.4. *Characterization of corrosion products*

XRD patterns of corrosion products formed on the two-bell surface and the iron clapper, fig. (7-a, b) and the results presented in tab. (2) revealed that the green corrosion products were atacamite, paratacamite ($CuCl_2.3Cu(OH)_2$) and malachite ($Cu_2CO_3(OH)_2$) incorporated with gypsum ($CaSO_4.2H_2O$). Whereas the corrosion products of the iron clapper were goethite ($FeO(OH)$) contains quartz (SiO_2) particles.

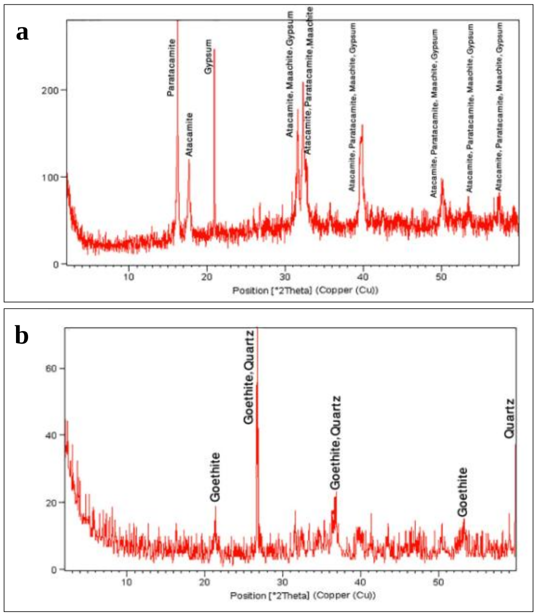


Figure (7) Shows XRD patterns of corrosion products a. formed on the bell surface, b. sampled from the iron clapper.

Table (2) XRD analytical results of corrosion products from the bronze bell and iron clapper.

Sample	Mineral Name	Chemical Formula	Intensity	Ref. Code
Bell surface	Atacamite	$CuCl_2.3Cu(OH)_2$	Major	02-0146
	Paratacamite	$CuCl_2.3Cu(OH)_2$	Major	19-0389
	Malachite	$Cu_2CO_3(OH)_2$	Minor	10-0399
	Gypsum	$CaSO_4.2H_2O$	Traces	00-01807
Iron clapper	Goethite	$FeO(OH)$	Major	01-081-0463
	Quartz	SiO_2	Traces	00-005-0490

4. Discussion

From metallographic investigations, it is observed that the alloy represents the tin bronze alloy. It consisted basically of a mixture of the α-Cu phase and the (α-Cu+ δ-Sn) eutectoid, in proportions depending on tin content and mold materials during casting. These latter affect the cooling rate, which in turn, influences the grain size and the nature of the α phase as well as the amount of the (α+δ) inter-dendritic eutectoid [14,20]. When lead is added to low-tin bronzes, typically castings, the lead does not alloy with the copper or tin and occurs as small globules throughout the structure [20,21]. It can be noted from EDS elemental mapping that the distribution of copper (Cu) and tin (Sn) indicate homogeneous matrix microstructure. According to XRF and EDS results, it can be concluded that the bell-making alloy was tin-bronze alloy. Bells were often made in antiquity of ternary tin bronzes consisting of about 20-25 wt % tin, 2-10 wt % lead, the remainder being copper, which considered the most important alloy for bell manufacture [13,20], Gadalla [4] was also confirmed that the chemical analysis of the typical ancient Egyptian bell was found to be 82.4 wt % copper, 16.4 wt % tin, and 1.2 wt % lead. Silicon and calcium which have detected by pXRF may be related to soil inclusions which incorporated with the corrosion products. The chloride content (3.072 wt % to 8.452 wt %) can be also attributed to the presence of soluble salts in burial environment. Based on the EDS results of the iron clapper, the high oxygen content (up to 42.03 wt %) could be attributed to iron corrosion products (typically iron (III) hydroxides). The Cu content may be referring to copper corrosion products, whereas the clapper is in direct contact with the bell body. It can be clear that the high carbon content showed in spectrum E and F (up to 10.60 wt %) could be attributed to epoxy resin which has been used for preparing the cross-section, not for the iron alloy. Because iron is seldom pure and minor levels of other components can introduce very different properties into the resultant alloy. The alloying element of greatest interest is carbon, which in concentrations ranging from 0 to 5 per cent greatly affects the properties of the different alloys formed [22]; wrought iron contains little carbon (not over 0.35 wt %); steel has a moderate carbon content (0.1-1.9 wt %); and cast iron has a high carbon content (2-4 wt %) [20]. Ancient iron can derive from fortuitous examples of meteoric iron, from native iron (telluric iron), or from smelted iron ores. Meteoric iron has provided early Egypt with no reliable and continuous supply source [12]. This usually contains Ni content approximately 10 wt % and is thus much harder and more difficult to work. The nickel content varies between 4 wt % and 26 wt % and can be easily detected, while there appear to be no ores that can homogenize nickel by direct melting [23]. Although Egypt has some remarkable iron ore deposits, but there is no evidence that these deposits were ever worked before the late or Graeco-Roman periods. The earliest shreds of iron smelting evidence in Egypt dated to about 580 B.C., where a large quantity of iron slag and some ore were found in the delta region at Naucratis and Defenna [24]. The results of XRD analysis showed that the two bells were subjected to being buried in wet soil rich in chloride ions and oxygen that were covered with a thick crust of green corrosion products incorporated with soil deposits. Quartz SiO_2 and gypsum $CaSO_4.2H_2O$ could be attributed to soil encrustations, while basic copper chloride compounds indicate the increasing content of chloride in soils which accelerates the corrosion rate through the destruction of the passive layer. Many researchers have studied corrosion processes of archaeological bronze objects in soil environment [25-30]. Those variable studies have led to that the most important parameters which have a severe degree of deterioration in the soil are soil pH, moisture content, temperature, acidification, salt content, and metallurgical characteristics of the metallic objects such as microstructure and composition. The bells are corroded in soil by forming a smooth noble patina (cuprite Cu_2O) on the surface that may become malachite due to the reaction between copper oxides and carbonate/bicarbonate dissolved in soil water. In addition, presence of chlorine ions in the burial environment penetrates the cuprite layer, forming a crystalline white layer of cuprous chloride (nantokite CuCl). This layer remains stable in the absence of both oxygen and moisture. During burial or after excavation, the artifacts are exposed to moisture and oxygen, cuprous oxide (cuprite) is produced by hydrolysis of cuprous chloride, and the hydrochloric acid will also be arisen by this reaction producing more cuprous chloride which promotes corrosion of the remaining metal. Cuprous chloride activity is increased when it is in contact with copper and a drop of water. It turns into a pale green layer in the phase of base copper chloride ($CuCl_2.3Cu(OH)_2$) paratacamite, then turns to atacamite, which damage the object and may change its shape [31-35]. Regarding the corrosion process of the iron clapper, it could be attributed to the galvanic corrosion which arises between the two metals (iron clapper and bell metal) differs in electrochemical potential in the presence of electrolytic solution. The less resistant metal (iron) becomes anodic and the more resistant metal (copper) becomes cathodic. Usually, the cathodic metal corrodes very little or not at all, while the anodic metal is highly corroded [36,37]. When iron corrodes during burial underground, an outer layer is found on its surface composed of a mixture of iron corrosion products (iron (III) hydroxide oxides). The second layer contains iron corrosion products in a lower oxidation state, usually magnetite. Goethite and magnetite are two common iron oxides that form during burial, with the latter forming when the level of dissolved oxygen is low. Heavily chloride-ions in soils can be guaranteed to create severe pitting or delamination of iron, often with severe loss, with or complete conversion to iron corrosion products [35,38,39].

5. Treatment Conservation

Depending on the examination and analysis results, conservation procedures were taken to remove the superficial encrustations/ deposits from the bell surface and preserve it against further attack. Compared with chemical cleaning process, the corrosion-mechanical cleaning is the most convenient and does not introduce chemicals into the artifact, which may compromise the shape of an object [22,34]. In the present case, the thick corrosion layers are like a support to the bell thin wall from both sides. The complete removal of these layers using chemical solutions will weaken the bell's structure or cause a significant loss of shape. In this case, the use of mechanical cleaning within brushes, scalpels and dental drilling machine is the preferred option to reduce the thickness of the hard-thick corrosion layers in a controlled way to reach a smooth layer that reveal the shape and modify the surface appearance. Active corrosion spots (the light green powdery eruption) were also mechanically cleaned by pointed needle under the magnifying glass [40]. The final treatment stage is to protect the bells against further corrosion; this can be achieved by treating the bells with corrosion inhibitor Benzotriazole 3 wt % in alcohol to stabilize the active corrosion areas. Then, a clear protective coating of Permalac (N-Butyl acetate) was applied by soft brush. This coat is developed in 1995 that is an air-drying lacquer in less than 5 minutes manufactured by Peacock Laboratories. It can be easily and completely removed by wiping the surface down with acetone [41-43], fig. (8). To slow down the process of deterioration, the two bells were housed in a microclimate polyethylene storage box with silica gel to control relative humidity. Silica gel should be checked and reconditioned regularly to be dry. The length of time silica gel will keep the desired RH before it needs to be changed, will depend on the box air exchange rate, the external humidity, and the quantity of gel [40,44,45].

Figure (8) Shows the two bells after the treatment processes.

6. Conclusion

High tin-bronze alloy is the preferred alloy for bells making which is containing about 22-24 wt % of tin, because it has given a pleasing sound quality when struck. Through metallurgical analysis of the bell manufacturing alloy, two-phased microstructure, the α-Cu phase and the (α-Cu + δ-Sn) eutectoid was observed with Pb small globules and scattered porosity. The iron clapper is completely transformed into corrosion products; therefore, it was difficult to identify its microstructure and elemental composition. Based on the corrosion results, the degradation of the two bells is due to aggressive/corrosive conditions of the burial environment, where soluble chloride and water content are the main corrosive features. Because the bell body is sandwiched between outer and inner corrosion layers supporting the bell wall, the cleaning processes were carried out to reduce the thickness of the corrosion layers without complete removal, in order to preserve the stability and integrity of the bell structure.

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