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ARCHAEOMETRIC CHARACTERIZATION AND CONSERVATION OF BRONZE PATINA ON ARCHAEOLOGICAL AXE HEAD IN MILITARY MUSEUM, CAIRO

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ABSTRACT

The archaeological community tried to study, protect and preserve artifacts because of its importance in representing the identity of civilizations. This research deals with a bronze axe head that was preserved in the military museum in Salah El-Din citadel in Cairo. In the first stage metallographic microscope was used to identify the surface shape and its fine details. Then, a scanning electron microscope with an energy dispersive unit was used to identify the elements and the type of alloy. The examinations and analysis showed that the axe was made of bronze alloy which is binary alloy essentially consists of copper and tin with other elements as impurities like iron and sulfur. Finally, corrosion products were identified using X-ray diffraction analysis as Paratacamite, Antlerite and Brochantite. After carrying out the necessary examination and analysis, mechanical and chemical treatments were carried out, sequentially. In the end, the axe was isolated using an acrylic coating (Paraloid B-72) to protect it against the ambient conditions.

KEYWORDS:
Archaeology; Weapon; bronze; Coatings; Conservation.

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التوصيف الأثري والحفاظ على باتينا البرونزي على رأس فأس أثري بالمتحف الحربي بالقاهرة

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الملخص

حاول المجتمع الأثري دراسة وحماية ووقاية القطع الأثرية؛ لأهميتها في التعريف بهوية الحضارات. تناول هذا البحث رأس فأس محفوظة في المتحف الحربي بقلعة صلاح الدين في القاهرة. في المرحلة الأولى تم استخدام الميكروسكوب الميتالوجرافي لتحديد شكل السطح وتفاصيله الدقيقة. بعد ذلك استُخدم الميكروسكوب الإلكتروني المزود بوحدة تشتيت الطاقة لتحديد العناصر الكيميائية ونوع السبيكة. أوضحت الفحوص والتحاليل أن الفأس مصنوعة من سبيكة البرونز، والتي تعد سبيكة ثنائية تتكون بشكل أساسي من النحاس والقصدير مع عناصر أخرى كشوائب مثل الحديد والكبريت. أخيرًا تم تحديد مركبات التآكل باستخدام التحليل بحيود الأشعة السينية مثل البارأتاكميت والأنتلريت والبروكانتيت. بعد القيام بعمليات الفحوص والتحاليل اللازمة، تم إجراء المعالجة الميكانيكية والكيميائية بالتتابع. في النهاية تم عزل الفأس باستخدام طلاء الأكريليك (بارالويد ب-٧٢) للحماية من الظروف المحيطة.

الكلمات الدالة:

الآثار، سلاح، برونز، طلاء، الحفاظ.

INTRODUCTION

Weapons made up a large part of the artifacts which date back to ancient Egypt civilization. It played a major role in the battles of ancient Egypt, and some opinions believe that these weapons developed at the beginning from the tools that the ancient Egyptian used for the purposes of daily life, and then they were modified with their function in wars.[1] By looking at the ancient history, we find that the axe was used by soldiers in various shaped, from the semi-circular shape to the duck beak shape.[2] In general, the axe was made through two methods. The first one is to be produced through segmented molds, which have a front and a back parts installed on each other. Then the molten metal is poured into it through a hole in it. The second method is to use the mold and hammering by pouring the molten metal into the mold and then hammering the piece to get it out in the desired shape. The bronze alloy consists mostly of copper (80-95%), tin (5-20%), and smaller proportions of other metals such as zinc and lead.[3] Bronze was the alloy used in the manufacture of weapons in ancient Egypt. Despite the availability of copper in the Egyptian lands, it was not suitable for use in the manufacture of weapons alone.[4,5] Because of the importance of these weapons and models in understanding the historical development that occurred in the ancient Egyptian army, it was necessary to preserve them from deterioration over time.

The conservation process takes place through different cleaning treatments and then isolation using chemicals, which are either corrosion inhibitors or coatings that are placed on the surface. The isolation aims to create a barrier between the surface of the axe and the surrounding medium and the various factors in it that may lead to more damage and corrosion, especially in the event of active corrosion due to the presence of chlorides.[6]

With regard to protective coatings for the surfaces of metal artifacts, over the past years a large number of them have been used. In the beginning, waxes, shellac, and vinyl resins were used, in addition to the acrylate group represented in Paraloid.[7] It consists of ethyl methacrylate and methyl methacrylate by 70-30%, and it dissolves in xylene, toluene, or acetone.[8,9] These coatings were used at different times and with several methods of preparation and conditions. Each of these coatings played a major role in protecting the pieces from further damage. These coatings were applied after carrying out the treatment and maintenance operations, including mechanical and chemical cleaning, and what is necessary for the object. However, over time, it was found that these coatings of all kinds

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1 Sabbahy L., *All Things Ancient Egypt: An Encyclopedia of The Ancient Egyptian World*, California, 2019.

2 Howard D., *Bronze Age Military Equipment, South Yorkshire,* Casemate Publishers, 2011.

3 Goffer Z., *Archaeological Chemistry*, Palastine, John Wiley & Sons, 2006.

4 Helmi F., Abu Ayana Y., and Omar S., Nano-*Copper Composite Conservation of An Egyptian Bronze Sacred Ibis Bird Statue: Case Study*, Mediterranean Archaeology and Archaeometry, 2019, 61-69.

5 Artioli G., *Scientific Methods and Cultural Heritage: An Introduction to The Application of Materials Science to Archaeometry and Conservation Science,* Oxford, OUP Oxford, 2010.

6 Wagle P., Simran T. and Aarti M., *Peelable Coatings: A Review, Progress in Organic Coatings,* 2021.

7 Dillman P., et al*., Corrosion of Metallic Heritage Artefacts: Investigation*, Conservation and Prediction of Long Term Behaviour, New York, 2014.

8 Cano E., Diana L. and David B., *Use of EIS For the Evaluation of The Protective Properties Of Coatings For Metallic Cultural Heritage*: A Review, Journal of Solid State Electrochemistry, 2010, 381–391.

9 Barat B. and Emilio C., *Evaluación in Situ De Recubrimientos Protectores Para Patrimonio Cultural Metálico Mediante Espectroscopía De Impedancia Electroquímica*, Ge-Conservación 8, 2015, 6-13.

suffer from common defects, such as loss of transparency due to interaction with the environment, as well as photo-oxidation,[10] biological and microbiological damage, cracks, holes, and separation from the applied surface because of that, new coatings were developed with nanomaterials to act in a better way.

MATERIALS AND METHODS

Optical Microscope (OM)

An optical microscope model (Leica DM500) located in the laboratories of the Faculty of Archaeology-Fayoum University- Egypt was used to examine the bronze patina with various colours of corrosion products.

Metallographic microscope

The metallurgical microscope located at the Tabbin Institute for Metallurgical Studies (TIMS) in Helwan was used to identify the structural composition of the samples taken to identify the method of producing them, whether by hammering, casting, or both methods together. This type of microscope is also used to identify the different forms of bronze patina that may appear in the sample. Metallographic microscope helps to determine the conditions that the axe was exposed to and then led to its damage, whether these conditions were environmental or occurred during the manufacturing, use or burial stage.

At this stage, a mold was made for the sample using Chemabox 150 (Chemicals for Modern Building CMB company - Egypt) by mixing the polymer with the hardener in a ratio of (2:1). The sample was fixed by placing it in a mold with a circular shape, then epoxy resin was poured and left to dry for 6 hours. After that, the samples were grinded and polished to be ready for examination.

Scanning Electron Microscope coupled With Energy Dispersive Unit (SEM-EDS)

A scanning electron microscope equipped with an energy dispersive unit model (FEI Inspect S 50-Netherlands Energy Dispersive X-Ray Spectroscopy EDS Attachment with Bruker AXS-Flash Detector 410-M-GERMANY) was used in the laboratories of the Tabbin Institute for Metallurgical Studies (TIMS) in Helwan to determine the elements present in the axe, in addition to the percentages of each element. Samples were used without any preparation.

X-ray Diffraction Analysis

The X-ray diffraction analysis was done at the Mineral Resources Authority (Geological Survey Authority) in Dokki – Giza. We used Secondary Monochromator, Cu-radiation (λ =1.542Å) at 45 K.V., 35 M.A. and scanning speed 0.04o/sec. were used. The diffraction peaks between $2\theta = 2^o$ and 60^o, corresponding spacing (d, Å) and relative intensities (I/I^o) were obtained. The diffraction charts and relative intensities are obtained and compared with ICDD files. This analysis was done by powder samples of corrosion products to specify the contents of axe's burial environment, mechanism of damage, and then determine the appropriate treatment method for it.

10 Artesani A., et al., *Recent Advances in Protective Coatings for Cultural Heritage–An Overview*, Coatings, 2020.

RESULTS

Examination by Optical microscope (OM)

Through Optical microscope a sample of corrosion products which was found on the surface of the axe was analyzed, a number of corrosion products were distinguished according to their distinctive colors, which were confirmed by subsequent analyzes.

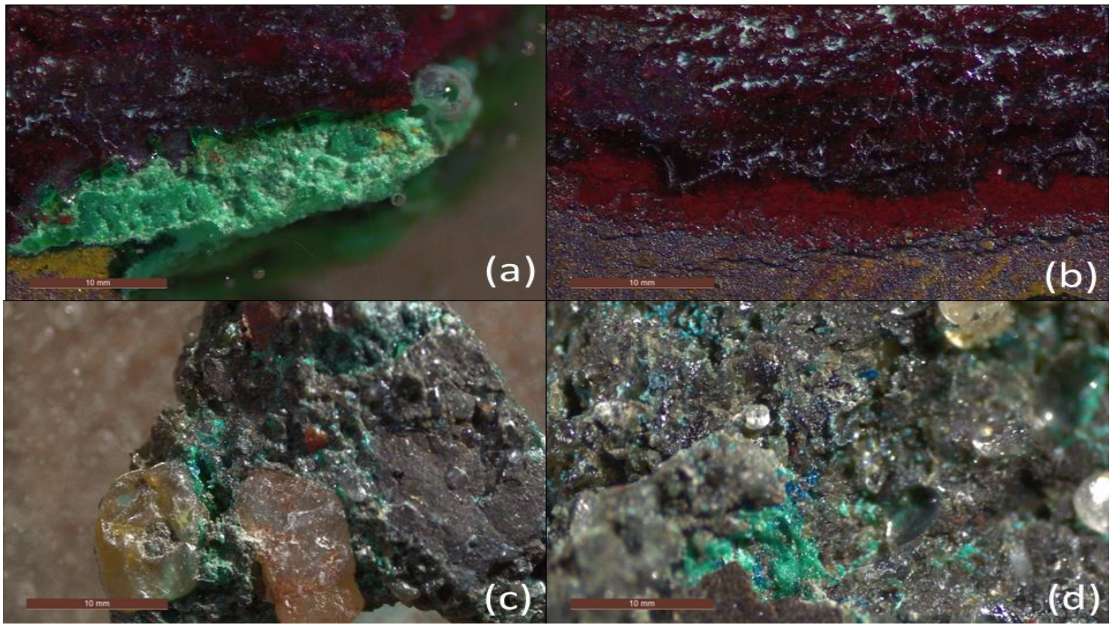

Figure 1 shows samples of corrosion products using optical microscope, (a) Paratacamite in a chalky green color, and Wustite (FeO) in a dark yellow color, (b) succession of layers of corrosion products in the sample and the presence of a layer of red iron impurities, (c-d) the overlapping of soil sediments with the Brochantite compound, which has a green color, and is produced due to the presence of sulfates.

Examination by Metallographic microscope

By examining a cross-section of a metal sample from the axe under a metallographic microscope with different magnifications, it was possible to show the structural composition of the sample. There were also micro-cracks through the alloy. Pitting corrosion can also be seen in separate places of the alloy which makes the alloy porous and weak.

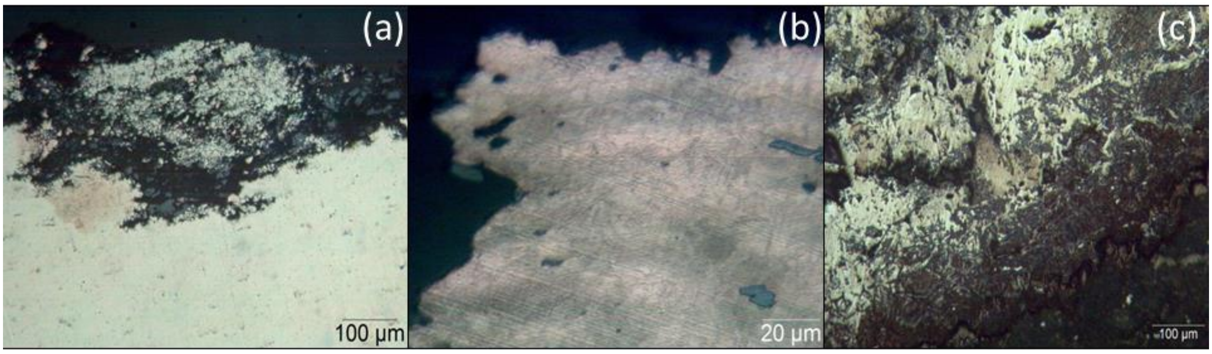

Figure 2 shows a metal sample from the axe using a metallographic microscope, (a) localized corrosion on the surface of axe, (b) the structure of the axe and pitting corrosion, (c) deep cracks inside the sample.

Examination and analysis by SEM&EDS

Three samples from the head of the axe were examined by a scanning electron microscope with an energy dispersive unit, with different degrees of magnification as shown in figure (3). The results showed the presence of pitting corrosion and deformation around the perimeter of the grains, as well as the presence of cracks throughout the body of antiquity, in addition to the obvious deformation of the metal surface.

Copper was found in sample (1) in percentage (60.99%) and tin (9.37%), in addition to the presence of chloride at a high rate of (24.29%), which is responsible for the presence of Paratacamite in the samples. The presence of sulfur in the sample was (5.32%), which may indicate the attack of chlorine and sulfur ions on the body of the axe, which explains the occurrence of significant damage in the future. In sample (2), the presence of copper and tin, which are the main components of the bronze alloy, were identified in the following proportions, respectively (83.74%), (13.84%), with less impurities of sulfur and chlorine, compared to the first sample. The results of sample (3) showed the presence of copper and tin in the following proportions, respectively (62.35%), (10.85%), with the presence of sulfur and chlorine also in small proportions as impurities.

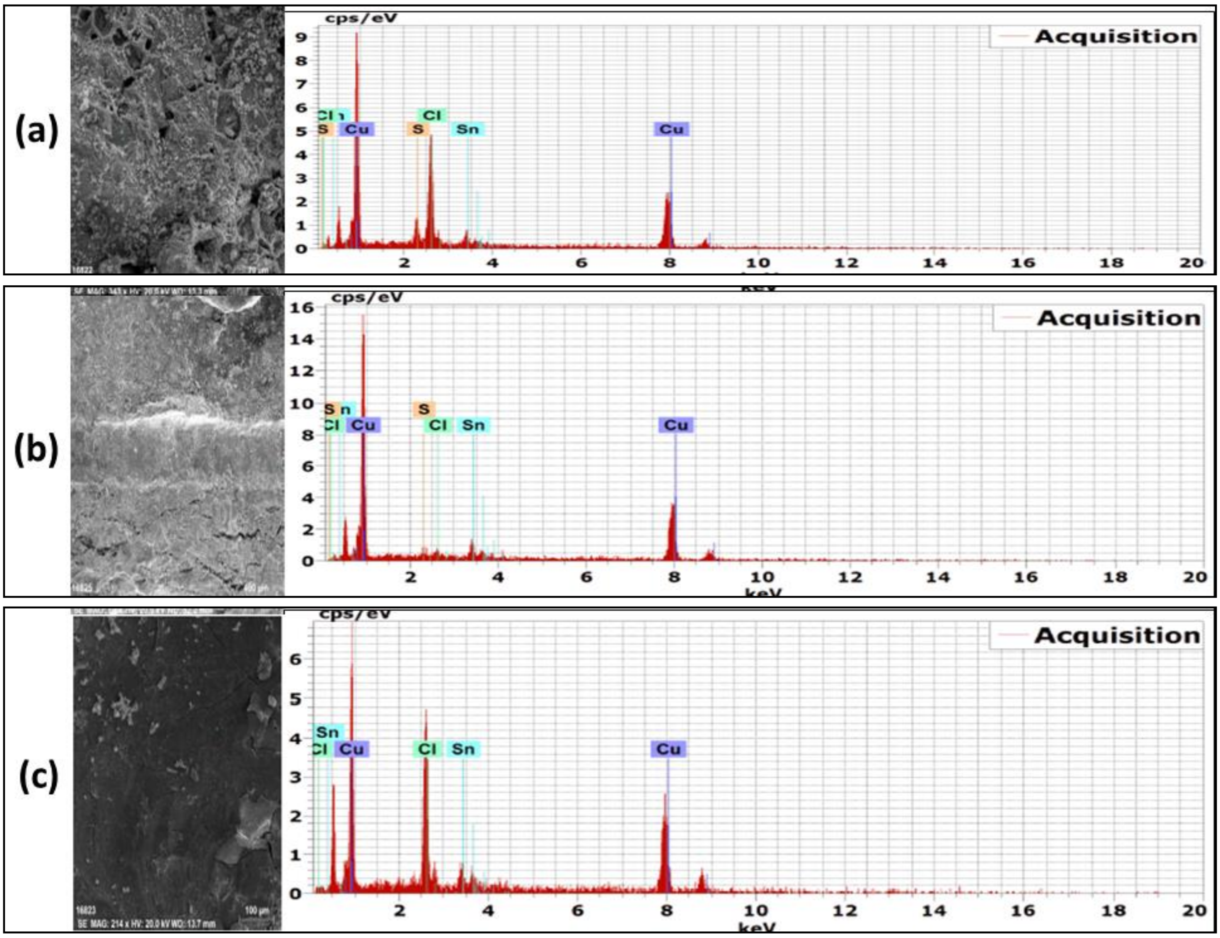

Figure 3 (a) shows the SEM & EDS of sample (1) and pitting corrosion appears well, (b) shows the SEM & EDS of sample (2) from the axe with rough surface, (c)shows the SEM & EDS of sample (3) surface with chloride impurities.

X-ray Diffraction (XRD)

Three samples of corrosion products from different parts of the axe were analyzed by XRD analysis, as shown in Figure (4). During the analysis of the first sample, the compounds that were found are Brochantite ($Cu_2SO_4(OH)_6$) with an emerald green color, Antlerite ($Cu_3(SO_4)(OH)_4$) with a light green color, and the Malachite ($CuCO_3.Cu(OH)_2$) with a color green, chalky green Paratacamite ($Cu_2Cl(OH)_3$), reddish-brown Cuprite (Cu_2O), Quartz (SiO_2), in addition to Wustite (FeO). Second sample showed the presence of Antlerite ($Cu_3(SO_4)(OH)_4$), Malachite ($CuCO_3.Cu(OH)_2$), Paratacamite ($Cu_2Cl(OH)_3$), Cuprite (Cu_2O), and Wustite (FeO). Via analyzing the third sample, Brochantite ($Cu_2SO_4(OH)_6$), Antlerite ($Cu_3(SO_4)(OH)_4$), Malachite ($CuCO_3.Cu(OH)_2$), Paratacamite ($Cu_2Cl(OH)_3$), Cuprite (Cu_2O) and Wustite (FeO) were found.

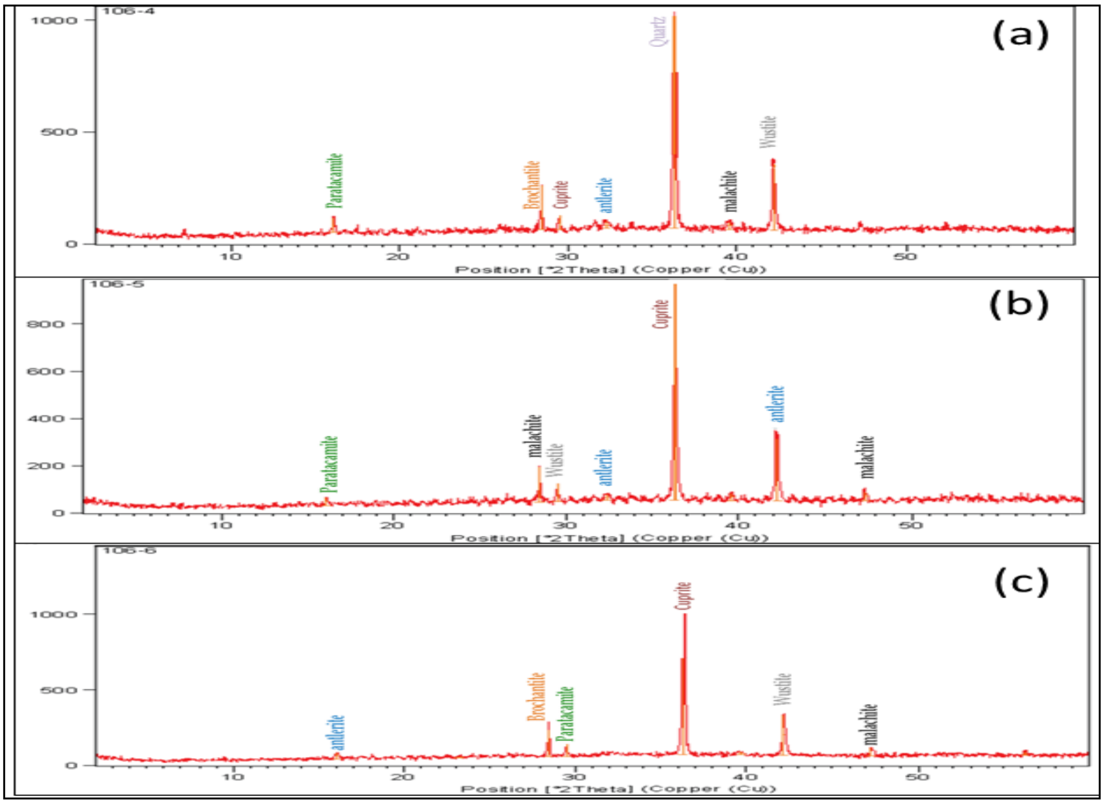

Figure 4 shows an X-ray diffraction analysis of the three samples, (a) the first sample, (b) the second sample, (c) the third sample, and the patterns of samples show approximately the same corrosion products.

CONSERVATION TREATMENTS

There are several conservation stages that can be applied on the object, but it depends on the object and its condition. These stages may be mechanical and chemical cleaning, reduction, consolidation and isolation. All of these different operations aim to protect the object stable.

Cleaning Operations

After the stage of examination, analysis, and identification of the surface features and the chemical compounds in corrosion products, the necessary cleaning operations of the axe head have been determined. Mechanical cleaning depends on simple tools such as brushes and scalpels, whereas, chemical cleaning depends on using basic solutions that do not negatively affect in the future.

Mechanical Cleaning

At this stage, scalpels and brushes made of brass were used, in addition to metal needles, in a circular motion with pressure on the axe with some force to help break the link between the layers of corrosion calcified and remove them. Magnifying lenses were used to complete cleaning operation well with focusing on not scratching the surface.[1] [1]

Chemical Cleaning

Mechanical cleaning operations were performed on the axe, but it wasn't sufficient. In the next stage, chemical cleaning operations were used, because the examinations and analyses proved that the axe head still retained its metallic state, and that it was strong, and even easy to remove without damaging it, this process was carried out as follows:

• Prepare a solution of Rochelle's alkaline salt, which consists of (sodium potassium tartrate) in an amount of 150 gm., with sodium hydroxide in an amount of 50 gm., in a ratio of 1:3.

• The head of the axe was immersed for 3 hours to soften the corrosion products.

• Through chemical treatment brushing the soften corrosion products were undertaken.

• Rinse the axe well with running water, then distilled water to remove the remaining trace of the solution and stop its effect.

• Drying the axe well by immersing it in successive basins of alcohol and ether to get rid of the remaining water droplets.

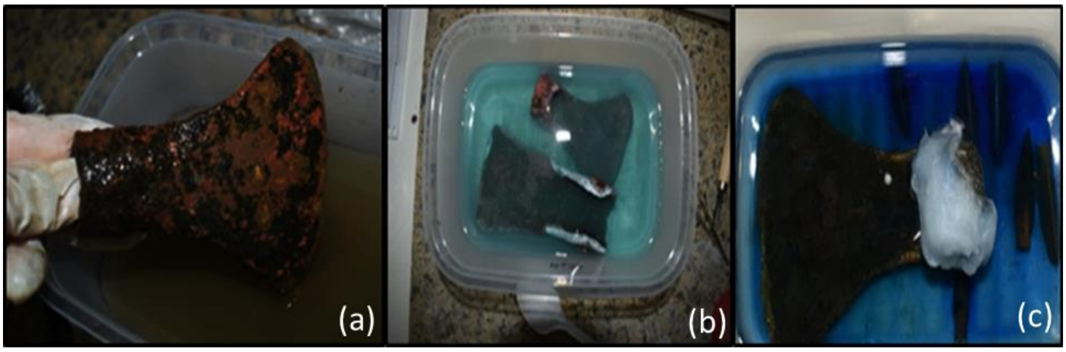

Figure 5 (a,b,c) shows conservation and treatment stages of the bronze axe.

Protective Coating

Organic coatings are widely used in the protection processes of cultural heritage artifacts. They act as barriers that prevent corrosion of the base metal due to the corrosive environment. Organic coatings keep the necessary resistance to weather, relative humidity, friction, and maintain the durability and aesthetic appearance of the piece. The efficiency of the organic coating depends on the mechanical properties of the coating system itself in addition to the pre-treatment of the metal

11 Paterakis B., *The Formation Of Acetate Corrosion On Bronze Antiquities*: Characterisation And Conservation, 2011.

surface, and the adhesion of the coating to the metal surface.[12] It should be noted that it is necessary to apply a primer coating that has good retarding and adhesion properties to the surface, and more than one coat provides good mechanical properties, pleasant color and transparency, and good waterproof and oxygen-proof properties.[13] We can improve the efficiency of the coatings if they are combined with corrosion inhibitors or wax.[14]

The viscosity of the coating is a very important factor in the success of the protection process. Increasing the viscosity of the coating leads to an increase in the thickness of the resulting film. This is eliminated via solvents with relatively slow evaporation rates.[15]

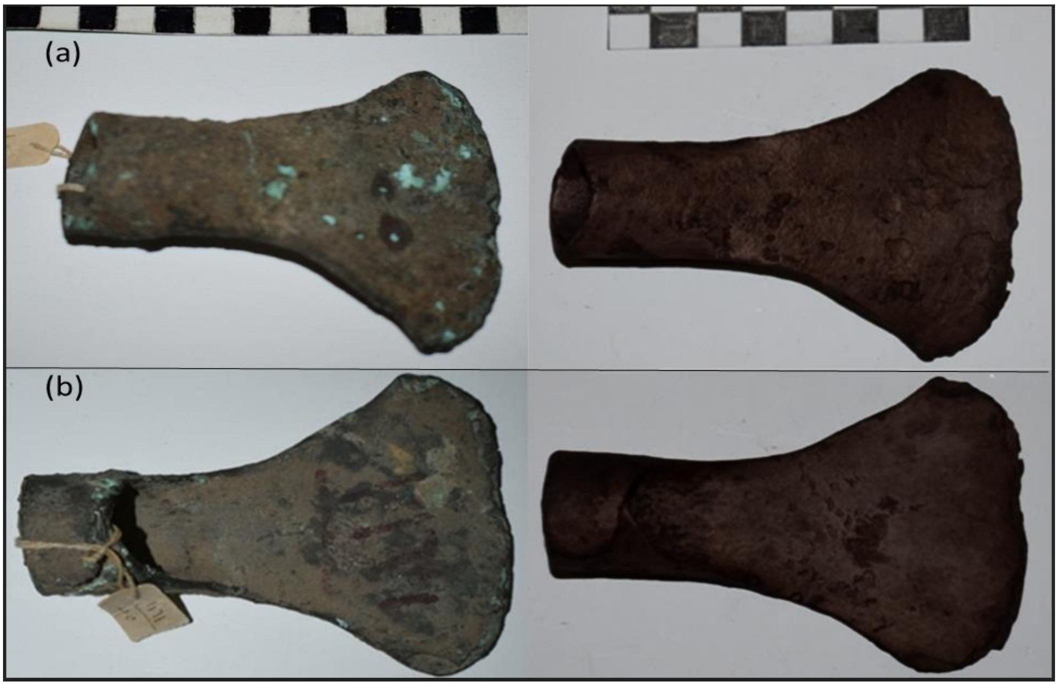

Figure 6 shows the bronze axe before and after the treatment, (a) front side of axe before and after conservation, (b) the backside of axe before and after conservation.

DISCUSSION

Through light microscope examination of corrosion products samples taken from the axe, a number of compounds were identified according to their characteristic colors. It was observed that there are clusters of Paratacamite ($Cu_2Cl(OH)_3$), which is caused by the presence of chloride ions in the burial environment. An interlayer of red iron inclusions was found, which explains the presence of Wustite (FeO) with dark yellow color, and this can be explained by the presence of iron as an impurity in the bronze alloy, or by the use of the same hearths used with iron.[16] Also, the Brochantite ($Cu_2SO_4(OH)_6$) which formed as a result of sulfates was identified, in addition to the Malachite ($CuCO_3.Cu(OH)_2$)

12 Popov B., *Corrosion Engineering: Principles and Solved Problems*, Amestrdam, Elsevier, 2015.

13 Paterakis B., *The Formation of Acetate Corrosion on Bronze Antiquities:* Characterisation and Conservation, 2011.

14 Ramírez B., Blanca P. and Emilio C., *An Overview of The Use of EIS Measurements for The Assessment of Patinas And Coatings In The Conservation Of Metallic Cultural Heritage*, 2019.

15 Lawson A., *Assessment of The Performance of Three Clear Coatings for Use In Heritage Conservation By An Oxygen Consumption Technique*, 2016.

16 Nathaniel L., et al., *Direct Evidence for The Co-manufacturing Of Early Iron and Copper-Alloy Artifacts in The Caucasus*, Journal of Archaeological Science 123, 2020.

which is resulting from the presence of carbon dioxide in the burial environment, intertwined with Quartz grains (SiO_2).

Through examination using a metallographic microscope, it was found that the axe suffered from pitting corrosion, which resulted due to the formation of localized cells in some weak areas (point defects) throughout the ingot.[17] It can be said that chlorides cause the appearance of pitting on the surface, where the surface is converted into soluble compounds and then removed from the surface afterwards.[18] As well as noting the spread of micro-cracks in some areas, which reflect the pressures that the axe suffered from, either through the stages of manufacturing and use, or during the burial stage.[19]

Subsequently, the surface morphology and alloy elements were identified through SEM-EDS. The head of the axe was made of the binary bronze alloy, which a miscible alloy is consisted of copper as a basic component and tin in a lesser proportion, in addition to other impurities. The literature mentioned the use of this alloy in the manufacture of weapons because of its high mechanical properties.[20] Three samples from the axe were taken to analyze them, it was found that the percentage of copper was in the samples, (60.9%), (83.7%), (62.3%), and tin (10.8%),(13.8%), (9.3%) respectively. Also we found relatively high levels of chlorine and sulfur impurities. The typical bronze of antiquity contains about 10% tin, although higher percentages of up to 16% have been found.[21]

Through the analysis of three samples of corrosion products, compounds formed due to sulfur ions such as Antlerite and Brochantite were found, in addition to the Malachite compound which is a result of carbon dioxide. The effect of the chloride ion was represented in the formation of atachamite and Paratacamite products, as well as the iron ions that led to form Wustite. These products are widely found in polluted environments.[22] Because the axe was buried for a long time in the soil, the corrosion products interfered with the soil sand grains represented by Quartz. The results also showed the presence of a Cuprite that formed a noble patina on the surface of the object.

After the necessary examination and analysis of the axe's head, conservation processes were carried out. Mechanical cleaning was used at first after making sure that the mechanical properties of the piece were sufficient, but it did not give good results. Chemical cleaning using Rochelle's basic salt was used to remove the remaining calcification, then the axe was exposed to baths of distilled water and then dried using alcohol and ether. After completing the drying process and as a final stage, the axe was isolated using Paraloid B-72 dissolved in acetone at a rate of 1-3% to protect it in the future from the ambient conditions.

CONCLUSION

This paper represents a study on a bronze axe through a number of examination and analyses. Optical, metallographic and scanning electron microscopes with energy dispersive unit were used. The results showed a large spread of pitting corrosion and micro-cracks throughout the axe body. In addition to a number of corrosion products that were identified by their distinctive colors and then verified via

17 Schweitzer P., *Fundamentals of Metallic Corrosion: Atmospheric and Media Corrosion of Metals*. CRC Press, 2006.

18 Sandell E., *New Materials for The Coating of Outdoor Bronze*, Columbia University, 2018.

19 Shoji T., *Factors Affecting Stress Corrosion Cracking (SCC) And Fundamental Mechanistic Understanding of Stainless Steels*, Woodhead Publishing Series In Metals And Surface Engineering, 2011.

20 Howard D., *Bronze Age Military Equipment. South Yorkshire*: Casemate Publishers, 2011.

21 Szczepanowska H., *Conservation of Cultural Heritage:* Key Principles and Approaches, Routledge, 2013.

22 Lawson A., *Assessment of The Performance Of Three Clear Coatings For Use In Heritage Conservation By An Oxygen Consumption Technique,* 2016.

EDS. The necessary processes of mechanical and chemical cleaning were carried out according to the condition of the piece. Finally the piece was brushed with Paraloid B-72 to protect from reactions with the surrounding environment. It is recommended to preserve the piece in appropriate temperatures and humidity to ensure the integrity of the axe.

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17- Szczepanowska H., Conservation Of Cultural Heritage: Key Principles And Approaches, Routledge, 2013.

18- Wagle P., Simran T. and Aarti M., Peelable Coatings: A Review, Progress In Organic Coatings, 2021.