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Chemical Characterization and Conservation of Bronze Coins Excavated from Tell Timai, El Sembelawen, Daqahleya, Egypt

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Abstract

Several archaeological coins were discovered in Tell Timai, Elsembelawen, Daqahleya. They were heavily corroded and in a bad state of preservation. Different corrosion products mixed with soil encrustation totally obscured the surface. The present paper aims to identify the morphology and composition of the alloy and corrosion products, perform a condition assessment and determine the best methods for treatment. Therefore, an analytical and characterization study on coin samples was carried out using X-ray diffraction analysis and portable X-ray fluorescence to understand the nature and composition of the alloy and patina. The results indicated the deterioration of the coins. X-ray diffraction analysis showed that the corrosion layers were mostly composed of chlorides, sulfates, and carbonates with some soil residues of gypsum and other compounds. X-ray fluorescence results showed that the coins consisted of ternary alloy [copper, tin, and lead].The composition of the coins indicated that several coins were high-leaded bronze, except one made from leaded copper. Finally, the results obtained help choose the best treatment and conservation methods. The conservation of the coins required removing the soiled deposits, encrustations, and corrosion layers covering the coins completely while preserving their integrity.

Keywords: Bronze coins; XRD; Portable XRF; Corrosion products; Leadedbronze;Leaded copper;Conservation

Introduction

Metals are one of the most used materials in all life activities throughout the ages. The uses of metals in Egypt started from the ancient Egyptian era and continued throughout the Coptic, Islamic and up to the modern times. Workers used metals such as copper, silver, gold, and alloys such as bronze, brass, and billon, to make their tools, weapons, jewellery and statues" as discussed by Al-Saad&Bani-Hani [1]".

Nearly all metals corrode after burial. Corrosion products can form thin, coherent layers or thick, disfiguring crusts that obscure the details of the object. They may protect the underlying metal or contain salts that cause further corrosion after excavation. These corrosion products are distinctly colored, some intensely so. They represent the first visual clue to the underlying metal composition and reflect the type of metal and the chemical composition of the soil.

Patina or thick corrosion crusts formed on copper, bronze, or other copper alloy artifacts may display complex products and structures. Some of those products or structural details may depend on the microstructure of the metal or the alloy attacked by corrosive agents after burial" as discussed by Ghoneim[2]".Soil is a complex environment with various parameters that influence archaeological materials in long term burial and causes changes in the appearance and the chemical nature of the buried objects "as discussed by Oudbashi, Scott& Selwyn[3,4,5]".

The conservation of ancient metals excavated from burial soil is an on-going challenge in **conservation** and restoration. The main aim of any conservation

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process is to improve the long-term preservation of the object by making it safe and pleasing for display while protecting its integrity "as discussed by Al-Sadoum&Abdel-Kareem[6]".

The archaeological science literature is rich regarding metallic object treatment. Cleaning, inhibition, consolidation, adhesion, reintegration and protection are common procedures in the field of metal conservation "as discussed by Lois, Vieira&Bosetto[7]".The post-depositional conservation and treatment of archaeological coins can have a dramatic effect on its composition. Ponting 1998[8]. In addition to the visual information preserved in the inscriptions, analytical data can also provide proof about the history and manufacture Oddy 1980[9]. The studied coins were excavated from the site of Thmuis in July 12, 2014 by an international team sponsored by the University of Hawaii led by Professor Robert Littman and Dr. Jay Silverstein. The coins date back to the Ptolemaic period. The ruins of the Greco-Roman Egyptian city of Thmuis were found at Tell Timai in the Delta region of Egypt near El-Mansoura. Thmuis is about half a kilometre to the south of Tell el Rub'a, the site of the ancient city of Mendes. Mendes was a dominant city in the Delta throughout Egypt's history, from the late fourth millennium until the fourth century B.C., partly because it was an important port. Apparently, the course of the Nile shifted in the fifth and fourth centuries, and Mendes, having lost its economic base as a port, was gradually abandoned for Thmuis, where the course of the river moved and a port developed "as discussed by Littman&Silvertein[10, 11]".

The Egyptian city of Ta-mawt ("new land"), known to the Greeks as Thmuis, is located amidst the salt-ridden soils of the Eastern Nile Delta within the Dakhaliya province. Today, the modern villages of Timai el-Amdid and Kafr el-Amir Abdulla Ibn Salam encroach upon the North-East and North-West borders of the tell; Kafr el-Amir is perhaps a development of the medieval Arab suburb of Thmuis. The tell itself stretches nearly one and a half kilometers in breadth and lies half a kilometer south of the smaller Tell el-Roba or ancient Mendes "as discussed by Morris[12]".

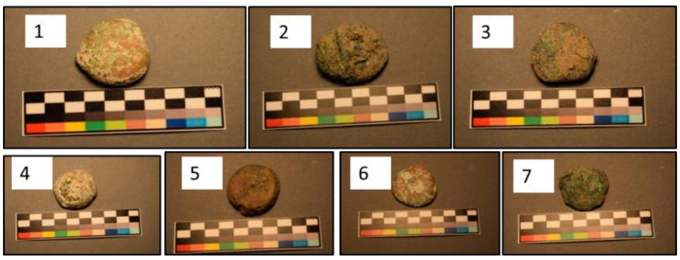
2.1. Materials and Methods

To characterize and determine the condition of the studied bronze coins and identify the different types of corrosion products covering the bronze coins, various investigation and analytical methods were carried out. The corroded surfaces of the objects were examined visually and microscopically by optical microscope using a Smart-Eye USB Digital Microscope at a magnification of1000X. Five samples of the corrosion products were obtained mechanically by scraping the corroded surface gently with a scalpel, then ground to a fine powder in an agate mortar and pressed into the specimen holder then mounted to be analyzed by the X-ray diffraction method. The corrosion products were selected to identify and determine their composition.

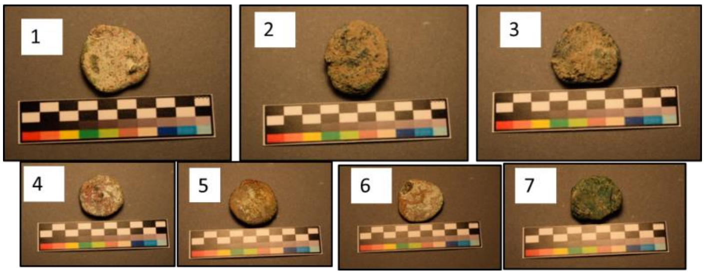
Samples were analyzed using Philips analytical X-Ray pw 1710 Diffractometer type. The operating conditions were: Cu tube anode, generator tension 40kv, and generator current 30 M.A. A portable x-ray fluorescence (XRF)Niton/ XLT 592 GKV, 8138 (USA), driven with software version 4.2E was used to identify the elemental composition of the coins nondestructively, as it provides qualitative and quantitative information about the elemental atomic structure of solid materials and identifies trace elements. The surface to be examined should be even and the same size as the fixed reference material calibrated by the device. The object and reference material are placed on the spectrometer in the same position and at the same angle with respect to the guide and the primary X-ray beam. [Kousouni et al. 2021][13].

2.2 Description of coins

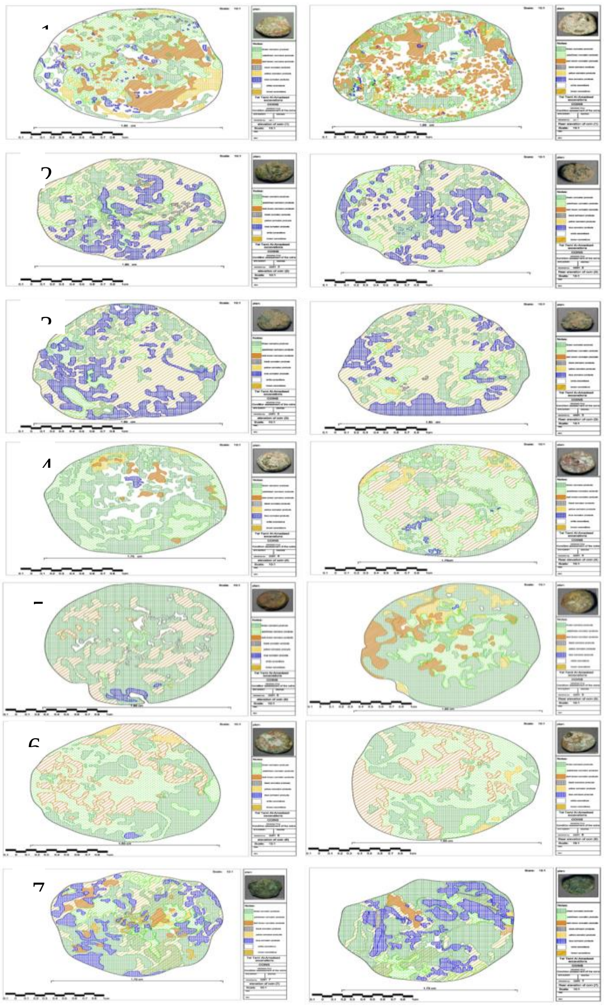
The collection consisted of seven bronze coins in a heavily corroded condition. Some were completely corroded; others were covered with an extremely disfiguring hard crust. This crust consisted of light–dark green and dark blue corrosion products incorporated with soil residues; some of them had small scratches among corrosion products



Fig(1) Showing the condition of the obverse side of the studied coins



Fig(2) Showing the condition of the reverse side of the studied coins



Fig(3) Documentation of the corrosion products of the studied coins using AutoCAD program. AutoCad was used to document the current condition of the coins to better understand the deterioration aspects and devise an interventive conservation plan.

3. Results and discussion:

3.1. Visual observation and microscopic examination

The results of visual observation and microscopic examination showed that the excavated archaeological bronze coins were covered with a thick layer of soiled deposits, encrustations, and corrosion products.

The effects of the processes of chemical alteration on the surface of the alloy were evident in the alteration crust formed on the mineralized surface. This chemical alteration might result from the degradation of the bronze coins during their long-term burial in soil. The excavated archaeological bronze coins were severely deteriorated with an extensive corrosion layer on the surface of the bronze coins. The surface of -the bronze coins was characterized by a rough corrosive surface with cracks. It was clear that various types of corrosion products in different colors, i.e., dark green and blue, with soil residues covered the coins.

3.2. XRD Analysis

The X-ray powder diffraction patterns obtained from five samples of different corrosion products covering the bronze coins revealed the presence of the following compounds; Malachite- $CuCO_3.Cu(OH)_2$,Azurite - $Cu_3(CO_3)_2(OH)_2$,Cerussit- $PbCO_3$,Anglesite-$PbSO_4$, Cuprous Chloride-CuCl. The results revealed that the components of the surface layer of the patina were green-colored copper (II) compounds incorporated with soil components covering a cuprous chloride and red cuprous oxide layer in contact with the metal of the alloy. Most of these compounds were of copper(carbonates) and lead (cerussite and anglesite),as detected by the portable x-ray fluorescence (XRF)as the main element of the alloy.

Malachite- $CuCO_3.Cu(OH)_2$ and Azurite - $Cu_3(CO_3)_2(OH)_2$ were identified as corrosion products in samples No. 2,3, and 7, representing a dark green layer and blue layer, respectively, Abdel-Kareem et al [14] mentioned that these compounds are the most corrosion products identified on copper objects. Malachite, or basic copper carbonate (CuCO3.Cu(OH) 2), is the product of the reaction between copper/copper oxides and carbonate/ bicarbonate anions dissolved in soil water. High concentrations of CO2 (or carbonate/ bicarbonate in soil) may lead to the formation of another isomer of basic copper carbonate, azurite "as discussed by Oudbashi, Naseri, Heidarpour&Ahmadi[15]".

However, azurite is less stable than malachite and may be converted to it in the presence of moisture through the loss of carbon dioxide "as discussed by Quintana[16]".

The normally protective film of cuprous oxide may fail in the presence of chloride ions because the chloride ion not only affects the relative stability of both oxidation states of the metal but also changes the speed of many electrode processes. The sources of chloride ions include groundwater, environmental sources, such as aerosols, and biological sources, such as sweat from humans.

Under the combined effects of ionic diffusion and electrical migration, chloride ions can penetrate the normally protective film of Cu_2O and react with the underlying metal to form a layer of cuprous chloride"as discussed by Macleod[17]". Relative humidity plays an important role in transforming the corrosion products from one type to another" as discussed by Saleh & Abu ElHassan[18]".Cerussite is formed from solutions with a high concentration of hydrogen carbonate ion (HCO_3^-), which usually results from the dissolution of calcium carbonate $CaCO_3$ by CO_2-rich water "as discussed by Franke&Mircea[19]".

$CaCO_3 + H_2O + CO_2 = Ca_2^{++} 2\ HCO_3^-$

Anglesite is formed in anaerobic soils.H_2S is produced by sulfate-reducing bacteria thatmight have penetrated the porous corrosion layers and corroded the Pb droplets to PbS. The formation of $PbSO_4$ could occur following an additional oxidation step "as discussed by McCann, Trentelman, Possley& Golding [20]".

3.3.Portable x-ray fluorescence (XRF):

The elemental composition of the objects was analyzed using portable x-ray fluorescence. The surface of the coins was cleaned mechanically before analysis, as chemical cleaning may alter the surface composition. The fact that the composition of the metal at the surface of a coin may not truly represent the exact composition of the bulk metal should be taken into consideration. Differences may be due to plating or selective corrosion. The results revealed that coins no. 1,2,3,4, and 6 were a leaded bronze alloy [Cu-Sn- Pb], while coin no. 5 was a bronze alloy [Cu-Sn], and coin no. 7 was a leaded copper alloy [Cu-Pb]. The qualitative and quantitative analysis of the coins is shown in table [1]. The composition of the studied coins is in agreement with coins from the same historic period. Surface enrichment with lead is considered, but the analysis of the bulk metal for comparison was not possible. The addition of lead to copper and bronze has been long in practice . Lead makes the alloys of copper easier to cast; as it, improves the fluidity of the alloy in the melt Scott [21]. Coins with similar compositions are abundant in literature Griesser et al. [22] investigated twenty selected coins with different corrosion states by neutron diffraction. These investigations confirmed a high lead content between 20 and over 30 % Pb and a typical tin content of up to 8 % Sn in the bronze alloys.

King et al. [23] analyzed a group of bronze coins by electron microprobe. The analyses confirmed a high lead content of up to 28%.Gouda et al. [24] investigated eleven damaged objects, including a piece of damaged coin displayed at the Egyptian Museum in Cairo by X-ray fluorescence. The results indicated that the coin was made of lead–bronze alloy, which contained Cu (66.49%), Sn(8.15%), and Pb(24.09%).

Table(1) showing the results of XRF analysis

Coin no.	Cu	Sn	Pb	Co	Fe	Au
Coin (1) obverse	41.82	2.24	53.47	0.15	1.77	nt
Coin (1) Reverse	34.59	2.52	85.44	0.21	3.34	nt
Coin (2) obverse	45.83	1.29	51.92	0.19	nt	nt
Coin(3)obverse	73.26	3.20	21.33	0.26	0.89	0.27
Coin(3) reverse	63.08	5.26	28.65	0.13	2.01	0.25
Coin(4) obverse	39.55	2.21	57.11	0.12	0.38	39.55
Coin(4) reverse	47.55	1.75	49.56	0.19	0.31	47.55

Coin(5) obverse	81.89	4.92	11.27	0.13	1.13	0.13
Coin(5) reverse	81.89	4.92	11.27	nt	1.19	nt
Coin(6) obverse	34.49	2.60	61.74	nt	nt	nt
Coin(6) reverse	44.29	2.51	51.98	nt	nt	nt
Coin(7) obverse	58.39	0.53	39.08	nt	0.57	0.18
Coin(7) reverse	59.68	0.66	37.98	0.11	0.20	0.28

3.4. Conservation processes:

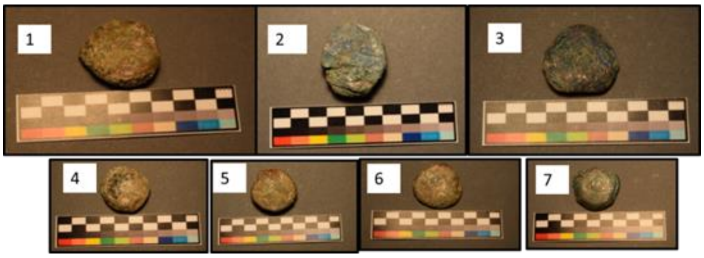
Conservation procedures should be carried out with caution and respect towards the integrity of the object 'as discussed by Pearson[25]'.After excavation, the damage of most metal artifacts may be aggravated rapidly due to environmental changes and corrosive compounds in the atmosphere. Thus, conservation treatments are crucial to prevent further damage and restore artifacts to their original form "as discussed by Sue [26]".It was important to develop a conservation plan according to the results of the characterization of the studied coins objects and their condition assessment. To succeed in establishing a conservation process that can be used effectively and safely in the conservation of these coins objects, various conservation methods were chosen according to their effectiveness and suitability to be applied.

3.5 Cleaning:

The main principle for choosing a suitable cleaning method is the long-time stability of the artifacts in the museum environment "as discussed by Viljus&Viljus[27]".

3.5.1 Mechanical cleaning:

A variety of brushes, abrasives, and hand tools are applied to clean the surface of coins and reveal the impeded inscriptions . Mechanical methods should be utilized whenever possible because they are controllable and do not involve aggressive chemicals, has less influence on the metallic surface. However, removing the corrosion crust bit-by-bit under the microscope is very time-and labor-consuming. Mechanical cleaning is the most convenient and does not compromise the shape of an object "as discussed by Cronyn, Scott, Gharib, Abdelbar, Megahed [28,29,30,31]".Apoultice consisting of ethanol: water at a ratio of 2: 1was applied on the surface to moisten and soften the encrustations to facilitate their easy removal "as discussed by Patel[32]".



Fig(10) showing the condition of the studied coins during mechanical cleaning

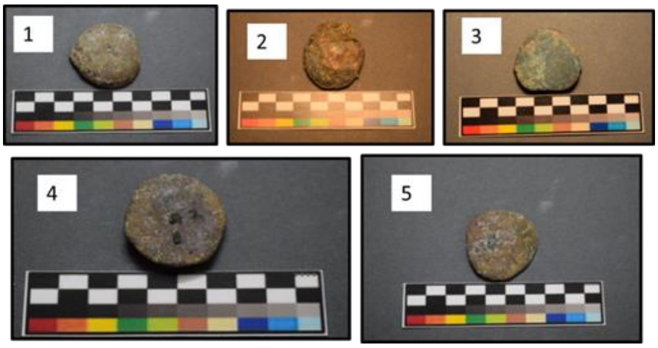
3.5.2 Chemical cleaning:

Coins no. 1, 4, and 6 were chemically cleaned using 5% wt/v. sodium sesquicarbonate solution. The water-insoluble copper chloride compounds reacted chemically with hydroxyl ions in the basic solution. As a result, copper oxide was formed. The solution was replaced until the chloride compounds were removed. A test for chloride ion was performed using silver nitrate test. The treatment goal was to get rid of any traces of chlorides that caused the bronze disease while keeping the patina and decorative patina intact"as discussed by Mahmoud&ElSerogy[33]".This treatment was followed by rinsing in distilled water and rinsing in alcohol.

The surface of coin no. 2 was treated with 2% sodium polyphosphate because of the presence of soil encrustations. However, the treatment caused color changes; thus, it was stopped. The surface of coin no3 was treated with 2%wt/v. trisodium phosphate. Cotton pads soaked in sodium tripolyphosphate solution were left in touch with the object. Then, the surface accretions were mechanically cleaned using a sharp scalpel.

For many years, sodium tripolyphosphate was employed in conservation to clean surfaces. It is an excellent sequestrant for magnesium, calcium, and other metal ions as discussed by Sharma &Kharbade[34].

Abdel-Kareem et al. [35] mentioned that not all conservators agree on specific conservation processes for cleaning of coins, they should understand of the consequences of using given products or applying certain techniques



Fig(11) showing the condition of the studied coins during chemical cleaning

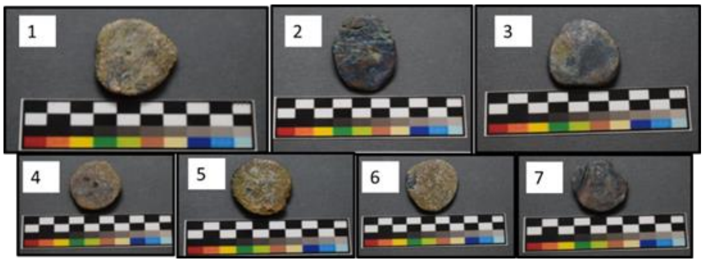
3.6. Stabilization:

Coin no. 3was stabilized using 3% w/v benzotriazole in ethanol solution to stop any active corrosion and prevent subsequent outbreaks. The coin was then submerged in ethanol to remove any remaining BTA.

The deposition of a passive film formed of an insoluble copper–BTA coating on active 'bronze disease' areas and on cuprite (Cu_2O) species acted as a protective barrier layer, protecting the bronze artifact from oxygen and moisture.

By inhibiting undesired surface reactions, (BTAH, $C_6H_5N_3$) is an efficient corrosion inhibitor for copper and its alloys. Since then, BTAH has kept copper surfaces from discolouring in both aerobic and submerged situations. It has been applied to preserve archaeological and historical artifacts as discussed by Finšgar&Milošev [36]. Finally, the coins were coated with 3% wt/v. Paraloid B72 in acetone.

fig(12) showing the condition of the obverse side of the studied coins after conservation



fig(13) showing the condition of the reverse side of the studied coins after conservation

The coins were kept in suitable microclimate in polyethylene or polypropylene boxes with snap-on lids and desiccant during all conservation procedures. Because the lid seal provides for the bulk of the air exchange rate, well-fitting lids are the most significant criterion to consider when evaluating the suitability of boxes for temporary or long term storage.

Desiccation is frequently used to cease or stop corrosion reactions. The most popular desiccant and buffering substance is silica gel. It is made up of porous, spherical silicate particles with a large surface area. It can absorb significant amounts of water vapour "as discussed by Rimmer, Thickett, Watkinson&Ganiaris[37]'.

4. Conclusion:

The current study emphasizes the importance of performing different analysis on excavated archaeological coins using X-ray diffraction analysis and portable X-ray fluorescence to understand the nature and composition of the alloy and patina prior to their treatment and conservation. The studied coins were excavated from the ruins of the Greco-Roman Egyptian city of Thmuis at Tell Timai in the Delta region of Egypt near El-Mansoura. The coins date back to the Ptolemaic period. The coins were documented using Autocad and a condition assessment was performed to know the extent of deterioration. The elemental composition of the studied coins revealed that the majority of the coins are a high leaded bronze alloy with lead content ranging from 11.27% to 61.47%, one bronze alloy, and one leaded copper. The addition of lead to bronze improves the alloy's fluidity in the melt, hence making it easier to cast intricate details. However, independently of the lead content in the bronze the corrosion process results in a strong relative enrichment in lead compounds in the corrosion layers as evident from the XRD analysis. Although the coins differed in their elemental composition, but they were severely deteriorated indicating that the degradation of the coins is due to aggressive/corrosive circumstances of the burial environment, where soluble chloride and water content are the main corrosive aspects, according to the corrosion results. Finally, to retain the stability and integrity of the structure, cleaning procedures were used to minimize the thickness of the corrosion layers without completely removing them, followed by stabilisation and coating. Mechanical cleaning is recommended whenever possible because it does not involve aggressive chemicals, which may alter the surface composition of the coins. Chemical cleaning should be performed with great care and applied locally, especially in areas of active corrosion. Stabilisation and coating is a necessity particularly if the storage conditions are not optimal.

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مخلص البحث:

تم إكتشاف العملات بتل تمي الأمديد بالسنبلاوين بمحافظة الدقهلية ويغطي العملات الآثرية مركبات صدأ ومخلفات التربة تكاد تخفي معالمها و يهدف هذا البحث إلي التعرف علي شكل و تكوين السبيكة و مركبات الصدأ ولذا تم إجراء مجموعة من التحاليل مثل التحليل بإستخدام حيود الأشعة السينية للتعرف علي مكونات مركبات الصدأ ومخلفات التربة والتحليل بإستخدام جهاز تفلور الأشعة السينية المحمول للتعرف علي مكونات السبيكة لفهم طبيعة و تكوين السبيكة و مركبات الصدأ و أظهرت النتائج التحاليل بإستخدام حيود الأشعة السينية إن طبقات مركبات الصدأ تتكون أساسا من الكلوريدات و الكبريتات و الكربونات و بعض مخلفات التربة و أظهرت نتائج التحليل بإستخدام تفلور الأشعة السينية أن العملات تتكون من سبيكة ثلاثية هي النحاس و القصدير و الرصاص و تتكون أغلب العملات من سبيكة البرونز التي تحتوي علي نسبة عالية من الرصاص و تتكون عملة واحدة من سبيكة النحاس و الرصاص وساعدت نتائج التحاليل في إختيارأفضل الطرق لعلاج وصيانة العملات الآثرية وتتطلب عملية علاج و صيانة العملات الآثرية إزالة مخلفات التربة و مركبات الصدأ التي تغطي سطح العملات الآثرية مع الحفاظ علي قوتها و صلابتها.