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AUTHENTICATION AND CONSERVATION OF SELECTED METAL OBJECTS EXCAVATED FROM AL-SEREIN, NEAR MAKKAH, SAUDI ARABIA

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ABSTRACT

This study aims to authenticate and to conserve some selected metal objects excavated from Al-Serein archaeological site, near Makkah, Saudi Arabia. The selected metal objects include rings, bracelets, fishing poles and coins. To success in developing and establishing conservation processes for these metal objects, scanning Electron Microscope (SEM) attached with energy- dispersive x-ray analyzer (EDAX) was used to identify and analyze the corrosion products on these metal objects.

For conservation of the coins it was necessary to remove the soiled deposits, encrustations, and corrosion layers covering the coins completely. For the other metal objects, rings, bracelets and fishing poles it was necessary to remove the soiled deposits, encrustations, and corrosion layers covering them with keeping the antiquities colors on the objects. In this study various cleaning methods within various conditions were used for cleaning the current metal objects according to their conditions and the aim of their cleaning. Mechanical cleaning, alkaline Rochelle salt, and alkaline dithionite reduction techniques were used in cleaning of the selected metal objects.

To evaluate the usefulness of the suggested conservation processes used in this study for cleaning the metal objects before, and after the cleaning processes were investigated by SEM with EDAX technique. The results have shown the effectiveness of the methodology. For the coins in the beginning all the corroded coins have to be treated with alkaline Rochelle salt. Then the silver coin was treated with alkaline dithionite reduction technique. Finally, after finishing the treatments, the metal objects should be rinsed, dried and followed by isolation with acrylic polymer.

The archaeological study of the decorations and inscriptions confirms that they date back to Ayyubid dynasty 569 to 649 H.D. (1147-1252A.D.). The presence of the name of the Abbasid Caliphate (Al-Musta'sim Billah Abu Ahmed), on the observe of the silver dirham and the name of Ayyubid Sultan (Al-Mansur Abu Bakr) on the reverse confirm that this dirham is date back to 640 to 656 H.D. (1242 – 1258 A.D.). The presence of the name of the Abbasid Caliphate (Al-Nasir li-Din Allah Abu Alabbas), on observe of the copper penny, confirms that this penny, is date back to 575 to 622 H.D. (1180 – 1225 A.D.).

KEYWORDS: Serein Excavation, Saudi Arabia, Ayyubid dynasty, Abbasid Caliphate, Corroded archaeological metal objects, soiled deposits, encrustations and corrosion layers, Corrosion products, conservation processes, mechanical cleaning, chemical cleaning, Alkaline Rochelle salt, Alkaline Dithionite Reduction Technique, SEM-EDAX.

1. INTRODUCTION

It is known that many trade routes were passing across many regions in the Arabian Peninsula in ancient times (Abdel-Kareem et al, 2016). These trade routes included sea routes such as the red sea and land routes such as silk road and incense route. In Islamic periods, Al-Serein town was a focal point of the trade road, pilgrimage (Hajj) road, and an important coastal road. All routes that left Yemen to Hijaz had to go through Al-Serein town. Also, Al-Serein was an important port between Egypt and Hijaz in Abbasid Caliphate and so increased in Ayyubid and Mameluke periods (Alzilay, 1986). So, there is a high probability to find archaeological objects in the excavations in this town that belong to various Islamic periods as a result of trade activities and pilgrimage (Hajj) trips to Mecca.

The long-term burial conditions in soil causes various corrosion morphologies in archaeological metals, from a thin corrosion layer to a completely corroded and mineralized artefact (Oudbashi, 2013) Corrosion products can form as thin, coherent layers, or thick, disfiguring crusts that obscure the details of the object. They may protect the underlying metal or they may contain salts that cause further corrosion after the object is dug out (Ghoniem, 2011). Archaeological metals, that contain a substantial amount of copper such as our case study, are easily damaged and corroded (Abdel-Kareem, et al, 2016, Viljus and Viljus, 2013). These metal objects were subjected to various corrosion processes resulting in different corrosion products that gradually alter their aspect, shape, nature, and resistance (Scott, 2002, Ullén, et al, 2004, Nord, et al, 2005, Ioanid et al, 2011, Al-Zahrani and Ghoniem, 2012, Real, et al, 2012, Oudbashi, 2013, Al-Saad and Bani-Hani, 2015, Abdel-Kareem, 2016).

The status of the excavated archaeological metal objects, the corrosion products, and thickness of their layers depend on various factors such as the chemical composition of the metal objects, manufacturing technology of the metal objects, and the environmental factors surrounding the metal objects in the burial soil such as humidity, salts, pollutants, microorganisms, etc. (Abdel-Kareem, 2016).

The identification of the corrosion products and characterization of archaeological metal objects is an essential requisite in order to acquire a better knowledge about the condition of ancient objects, corrosion processes and conservation treatment or preventive procedures for long-term, stable preservation (Ghoniem, 2011). Many efforts have been done to investigate, understand, and analyse the corrosion products on metal objects and to explain the mechanisms of the corrosions of the metals (Ingo, et al, 2002, Ingo et al, 2004, Ingo, et al, 2006, Campanella et al, 2007, Beck et al, 2008, Mousser, 2011, Rodrigues et al, 2011, Abdel-Kareem et al, 2011, Oudbashi, 2013, Sandu, et al, 2008, Wan et al, 2015, Kousouni et al., 2018).

Conservation of ancient metals excavated from burial soil, is still an ongoing challenge in the field of conservation and conservation science. The main aim of any conservation processes to an object is to improve the long-term preservation of the object by making it safe and pleasing for display (Kotoula and Kyranoudi, 2013, Abdel-Kareem, 2015). The most common conservation processes of a metal object include documentation, mechanical cleaning, chemical cleaning,and other cleaning methods such as laser cleaning, stabilization, isolation, restoration, exhibition, and storage (Koh, 2006, Doménech-Carbó et al, 2009, Viljus and Viljus, 2012, Novakovic et al, 2013, Dillmann et al, 2013, Abdel-Kareem et al, 2016). Any conservation process should be started with a complete examination and documentation of the object (Abdel-Kareem et al, 2011).

Cleaning is usually the first step of many processes in conservation work. It is one of the most difficult operations undertaken when conserving metal artifacts (Novakovic et al, 2013). Any cleaning process must be carried out with great respect and consideration of the original object's form, function, and material (Abdel-Kareem, 2016). For this reason, efforts should be done to find and develop new techniques and methods which will be more suitable for cleaning the corroded archaeological metal objects. There are various common methods used in cleaning of metal objects such as mechanical cleaning, chemical cleaning, alkaline dithionite galvanic cleaning, and electrolytic reduction cleaning (Abdel-Kareem, et al, 2016, Degrigny, 2007, Ghoneim and Megahed, 2009, Watkinson, 2010, Costa, 2015). Many methods are developed to conserve metals such as laser cleaning that is now becoming an accepted and important technique in conservation of archaeological metal objects (Abdel-Kareem et al, 2011, Hrnjić, 2015, Abdel-Kareem et al, 2016).

However, the new techniques are not always suitable and accepted for conservation of all materials such as metal objects excavated from soil with thick layers of crust and corrosion products. Also, sometimes the new techniques are not available in the conservation laboratories or in the archaeological sites. So, it is important to select available methods that can be applied easily and safely to conserve these types of archaeological metal objects. The most important requirement that should be considered is that the selected methods should not damage the surface of the metal objects and should be applied carefully to prevent any side effect of the misuse of any conservation technique. For example, the mechanical cleaning can be effective and useful in cleaning the thick layers of soiled deposits, encrustations, and corrosion products covering metal object, but this process should be stopped before touching of the fresh surface of the metal object. If the mechanical cleaning was done until the surface of the decoration of the metal object was completely revealed, the surface of the metal object, most probably, will be damaged. Also, not all chemical cleaning methods are suitable to clean metal objects. Some chemical methods used in cleaning metal objects can react with the metals and deteriorate them. So, any method that will be suggested to conserve the metal objects should be justified and applied with controlling all conditions.

Saudi Arabia has many important archaeological sites that belong to all various historical periods. In the last two decades, General Commission for tourism and national heritage in Saudi Arabia has increased its attention for archaeological excavations in various sites. One team of researchers from Saudi General Commission for tourism and national heritage has begun to excavate in Al-Serein archaeological site, near Makkah, Saudi Arabia. This team has found a lot of archaeological objects includes, pottery, stones and metal objects. The major finding in this excavation was metal objects that contain 9 coins, women jewelry (such as rings, anklets and bracelets), and fishing poles.

The key to the survival of these finding objects for future generations is the success in achieving an acceptable conservation processes suitable to preserve the historical, cultural and aesthetic values of these metal objects. Therefore, the current research should be helpful for conservators and interested scientists to insure that these objects are available for future generations.

This study aims to authenticate and to conserve some selected metal objects excavated from Al-Serein archaeological site. The studied metal objects contain 9 coins, bracelets and fishing poles. They were discovered in the first season of the project excavation works in the year 1437 H. / 2016 A.D. This excavation has done by the Saudi archaeological team under supervision of Dr. Awad Al-Zahrani. This study describes the conservation processes for conservation of the selected metal objects. To success in stablishing suitable and effective conservation processes, it was necessary to estimate the condition statues of the studied metal objects and their characterization. Various methods were used to remove the soiled deposits, encrustations, and corrosion layers covering the selected archaeological metal objects. More of the conservation processes had been done to reveal the surface of the the coins with their decorations, inscriptions, and symbols. Also, the study aims to authenticate the conserved coins according to the revealed writings and descriptions on the surface of these coins.

2. EXPERIMENTALS

2.1. Description of the studied metal objects

This study is carried out on corroded archaeological metal objects discovered from under soil at Al-Serein archaeological site, near Makkah, Saudi Arabia (see Fig. 1). The selected metal objects are 9 coins, rings, bracelets and fishing poles (see Figs. 2-3).

Figure 1: A) Map of Saudi Arabia, B) the position of Al-Serein town on the map of Saudi Arabia

They were discovered in the first season of the project excavation works in the year 1437 H. / 2016 A.D. This excavation has done by the Saudi archaeological team under supervision of Dr. Awad Al-Zahrani. Al-Serein was a small village near Makkah and was an important Port in Abbasid Period.

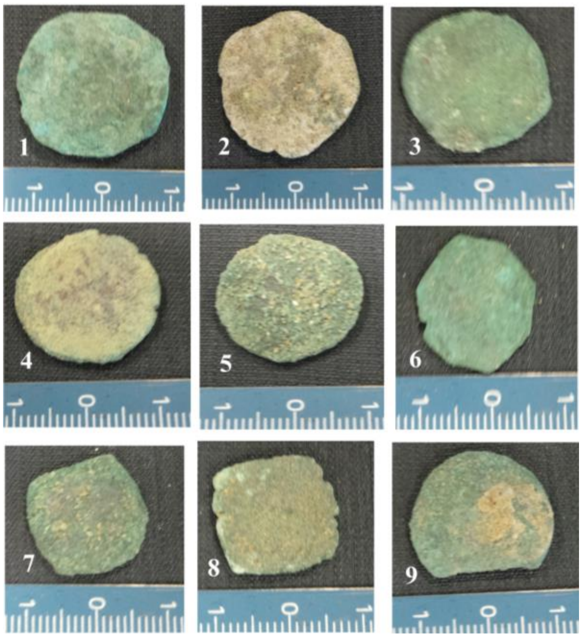

Figure 2: Photos show the corrosion products on the studied coins and their condition.

Figure 3: The corrosion products on some of the excavated metal objects that include rings, bracelets and fishing poles.

2.2 *The characterization and status of the studied metal objects*

To characterize and estimate the status of the studied metal objects and to identify types of the corrosion products covering these metal objects, various investigation and analysis methods were carried out on the selected metal objects. The metal objects were investigated visually and microscopy. The Scanning Electron Microscope with Energy dispersive X-ray Analysis (Model JEOL JSM-6510LV, voltage 30 kV) has been used according to Abdelkareem, 2015 to examine the surface morphology of the selected metal objects. EDAX analysis, was used as a non-invasive method to estimate and identify types of the corrosion products covered with a layer of corrosion products (in different colors, black, light blue to blue-green) covering surfaces of the selected metal objects.

2.3 *Conservation Processes*

Although that the results obtained by Abdel-Kareem et al, 2016 confirmed that laser cleaning was very effective and suitable to clean coins excavated from under soil in Saudi Arabia, It was not able to apply it in cleaning of these metal objects. This is because it is not available in this time in conservation laboratory, General Administration of Museums, Antiquities and Museums Branch, Saudi Commission for Tourism & Antiquities, Riyadh, Saudi Arabia. In the same time it was necessary to remove the corrosion products from the coins and the other selected metal objects to reveal the inscriptions and decorations to help archeologists study these coins and the other selected metal objects. So, it was important to establish a conservation plan according to the results of the characterization of the studied metal objects, their status, and the available facilities available. To succeed in establishing conservation process that can be used effectively and safely in conservation of these metal objects, various conservation methods were chosen according to their effectiveness and their suitability to be applied in the conservation laboratory, Saudi Commission for Tourism & Antiquities, Riyadh. According to the condition of the metal object and the aim of the treatment it was chosen the conservation processes that can be effective and achieved good results. Various conditions such as various periods and different concentrations for each method were used according to the aim of the conservation treatment.

2.3.1. Surface Cleaning

All the selected metal objects have been cleaned mechanically before any other treatments. The mechanical cleaning was done according to the conservation rules to remove the crusts and corrosions. Mechanical cleaning was applied to remove the superficial deposits/encrustations in a controlled and minimally obstructive way and then to reach a smooth layer which preserves the detail and shape. Features of interest included surface inscriptions and manufacturing details. The cleaning procedure involved removing the soft smooth corrosion and soil residues using mechanical tools, for example by brushes, spatula and scalpels. The mechanical cleaning was done carefully to make sure not to touch the original surface of the metal objects.

2.3.2. Treatment with Alkaline Rochelle salt

The selected metal objects that include rings, bracelets, fishing poles and 2 coins were chosen for the chemical treatments. The other coins were kept without treatment for any future studies. After removing the loosely corrosion products and crusts from the surface of the selected metal objects and coins (by using the surface cleaning methods), they were cleaned chemically using alkaline Rochelle salt. Alkaline Rochelle salt was prepared by dissolving 50gm of sodium hydroxide NaOH in every liter of cold water. Then 150 gm of sodium and potassium tartrate was added to this solution and was completely dissolved by good stirring. The prepared solution was stored in bottle until its use (Abdel-Kareem, 2015). The metal objects were immersed in consecutive bathes of alkaline Rochell salt solution (see Figs. 4-5). The period of each bath depends on the amount of the corrosion products that covers the metal object. The bath was changed when the color of the bath became blue.

During the immersion of the metal objects in the Rochel salt solution, the metal objects were brushed gently using a tooth brush to help separate the corrosion products from the surface of the metal objects. After the treatment with the Rochell salt, the metal objects were washed and cleaned with soft brush in distilled water. Then, the treated silver coin only was polished and gently rubbed with sodium bicarbonate. Cleaning with sodium bicarbonate is effective because its particles is not hard enough to scratch the metal surface, but it is still abrasive enough to remove the softened corrosion products. All cleaned metal objects were rinsed well in non-ionized water for more than 48 hours to remove any effects of treatment solutions. After that, they were dried and kept until the testing.

As the investigation results showed that there is no decorations and inscriptions on the studied metal objects except coins so the previous treatment was enough to rings, bracelets and fishing poles. But for the coins as the results showed that there is a necessity to reveal the inscriptions and decorations from the surface of the coins so other treatments were carried out. The results show that one of the coin is silver coin and the other is copper coin. For the copper coin, the visual examination of the coin revealed that it was covered with a hard superficial crust of corrosion layer. So that we treat the hard crust locally with sulfuric acid in very low concentrations and rinsed well in distilled water. Then we repeat the treatment with alkaline Rochelle salt on the coin until the surface became good and revealed the inscriptions on the coin surface. Then, it was rinsed well in non-ionized water for more than 48 hours to remove any effects of treatment solutions. After that, it was dried and kept until carrying out any other treatment. For the silver coin it was treated with Alkaline Dithionite Reduction Technique.

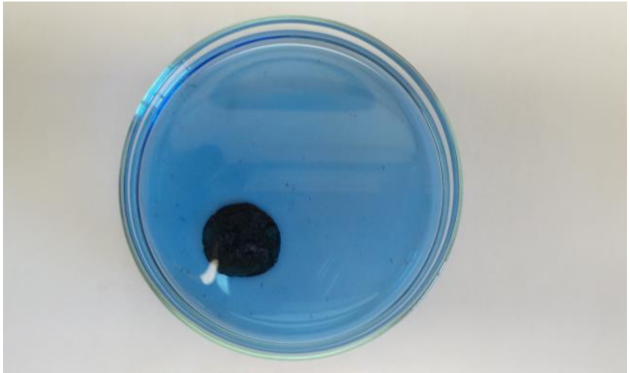

Figure 4: Treatment of coins with Rochelle salt

Figure 5: Treatment of rings, bracelets and fishing poles with alkaline Rochelle salt

2.3.3. Treatment with Alkaline Dithionite Reduction Technique

The Dithionate solution was prepared according to Abdel-Kareem 2015. This method was used only for cleaning of the silver coin. Then the coin was immersed in a 10% hydrochloric acid solution for 10 minutes in order to remove any remaining calcifications. Then the coin was rinsed well with running water. Then, the coin was immersed in dithionate solution in a closed glass bottle. The glass bottle was closed and shook well to ensure that the solution was mixed well and that it covered the coin surface. After nearly one hour, the coin was taken out and rinsed well with water to remove any remaining corrosion products from its surface. As the cleaning degree was not enough, so that the dithionate solution was changed and the process was repeated again.

After repeating this process many times, the coin was taken out and washed well with distilled water. Then, it was polished with sodium bicarbonate and rinsed well in non-ionized water for more than 48 hours to remove any effects of treatment solutions. After that, it was dried and kept until carrying out any other treatment.

2.3.4. Isolation of the metal surface (Stabilization of the future deterioration)

To inhibit further corrosion, the metal objects were degreased through swabbing with acetone and inhibited with 3% on Benzotriazole in ethanol by brush. The metal objects were given three coats, one hour between each application to be air-dried. Finally the metal objects were coated with a protective coating of Paraloid B-44 5% in ethanol (Scott, 2002, Ghoniem, 2014). Paraloid B-44 has good properties such as flexibility and its Tg is 60ºC that makes it suitable for the atmosphere in storage rooms in the national museum in Riyadh that where the objects will stored. The coating was applied in 3 layers. In between each application the film was allowed to dry and polymerize for 8 hours. The aim of this treatment to isolate of the metal objects from the environment condition surrounding the objects in the store room, while their storage in the museum.

2.4 Evaluation of the tested conservation processes

For evaluating the conservation status of the conserved selected metal objects, they were investigated before, during, and after the conservation processes visually and microscopy. Also SEM-EDAX was used to investigate the metal objects. All investigation methods were carried out as described above.

3. RESULTS AND DISCUSSION

3.1 Characterization and the status of the excavated archaeological metal objects

The results of visual observation and the microscopic examination showed that the excavated archaeological metal objects were found covered with a thick layer of soiled deposits, encrustations, and corrosion products. The results showed that the excavated archaeological metal objects were badly deteriorated and an extensive corrosion layer impeded all decorations and inscriptions on the surface of metal objects. This layer has different features. In some parts it is continuous, most even, and less thick, in other parts the surface is covered in holes, where the soil residues have the color of the corrosion products and is interposed with them. The corroded metal objects were covered with heterogeneous thick layer of crust in different colors and corrosion products coexisted with soil particles. Their aspects were so distorted that no detail of the original surface could be retrieved. The corrosion layer had a nearly composite structure, including metallic remains, mineralized, metallic, and insoluble phases, and products formed from the interaction between soil components, metal corrosion compounds, and soil particles. This might be resulted from the degradation of the metal objects that occurred during their long-term burial in soil. The surface of the tested metal objects characterized with a rough corrosive surface with cracks and pits. It was clear that there were various types of corrosion products in different colors: dark green, light green, greenish blue, and metallic gray blackish surfaces covered with soil residues (see Figs., 2-3).

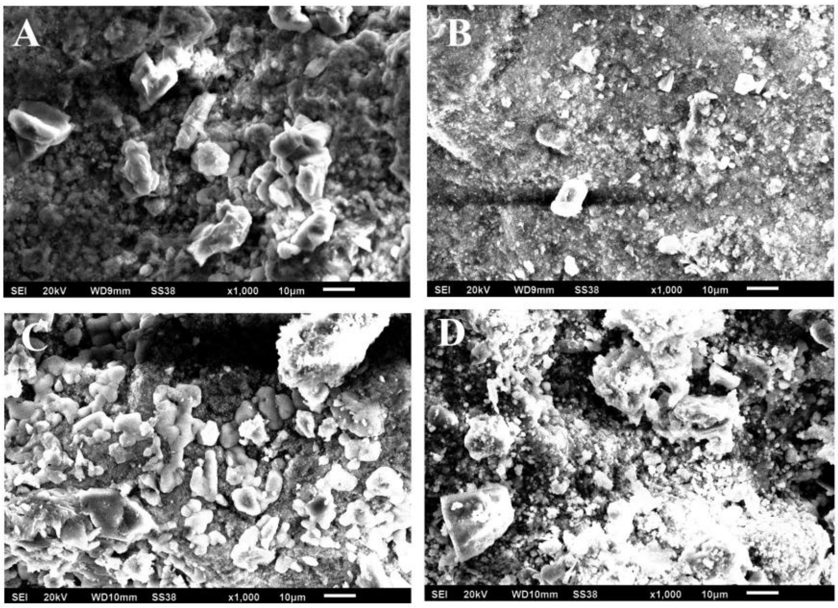

Figure 6: SEM images of the excavated archaeological coins before any treatment, (A coin 1, B coin 2, C coin 3, D coin 4). The morphological structure of the coins show that the crystalline and non-crystalline corrosion products formed an ugly and inhomogeneous surface.

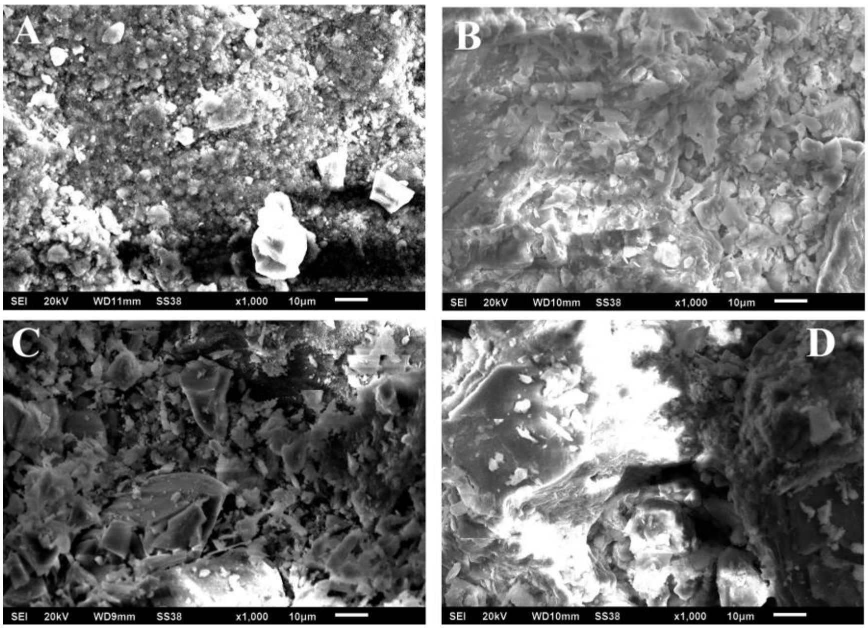

Figure 7: SEM images of the excavated archaeological metal objects before any treatment, (A ring, B anklet, C bracelet, D fishing pole). The morphological structure of the metal show that the crystalline and non-crystalline corrosion products formed an ugly and inhomogeneous surface.

The results of SEM morphological examination for the metal objects before the conservation processes showed that the morphological structure of the metal objects is characterized with raw hard crust and corrosion layer which was due to the corrosion produced on its surface. This layer of corrosion products mixed with soil relics that covered its feature and formed an ugly and inhomogeneous surface (see Figs 6-7).

The results of EDAX analysis of the excavated archaeological metal objects before cleaning are shown in tables 1-4. The results of the EDAX analysis for the corrosion layer of the metal objects indicated that contaminated elements such as O, C, Al, Si, S, Cl, K, Ca, and Fe were detected. The results show the presence of Cu, O, and C as major elements, while Al, Si, S, Cl, K, Ca, and Fe as minor elements. These results confirm that the corrosion layer consists of mainly copper corrosion products. The results indicate that the main substrate of the metal objects is not clear as these corrosion products are common present on copper metal objects, copper-alloy metal objects such as bronze and billon alloys (See the results obtained by Abdel-Kareem, et al, 2015, 2016, Ghoneim, 2011).

Table 1: The average of (wt. %) of the detected elements in the studied coins before the conservation

Element	Coin 1	Coin 2	Coin 3
C	10.09	12.94	6.97
O	35.76	39.06	37.24
Na	3.58	-	-
Mg	2.07	0.87	5.94
Al	2.74	1.98	1.90
Si	7.04	11.45	5.11
S	0.48	0.45	0.43
Cl	10.43	4.27	11.55
K	0.56	0.36	0.38
Ca	1.54	2.54	0.96
Fe	1.21	1.15	1.35
Cu	24.49	23.91	28.19
Zn	-	1.01	-
Totals	100	100	100

Table 2: The average of (wt. %) of the detected elements in the studied coins before the conservation

Element	Coin 4	Coin 5	Coin 6
C	6.30	6.04	6.29
O	32.56	39.09	38.11
Na	8.56	-	-
Mg	2.69	4.33	3.14
Al	2.09	2.94	3.50
Si	4.92	8.25	9.31
S	0.61	0.61	-
Cl	14.68	9.76	8.45
K	0.40	0.56	1.15
Ca	1.47	1.41	1.24
Fe	1.46	1.85	2.33
Cu	24.07	25.15	26.49
Totals	100	100	100

Table 3: The average of (wt. %) of the detected elements in the studied coins before the conservation

Element	Coin 7	Coin 8	Coin 9
C	9.01	7.39	6.65
O	38.04	37.27	43.09
Na	0.23	-	-
Mg	4.14	2.90	1.32
Al	2.03	2.07	2.68
Si	4.84	8.01	13.23
S	0.36	-	0.35
Cl	13.00	10.65	4.92
K	0.43	0.55	0.36
Ca	0.79	1.19	1.31
Fe	1.13	1.20	1.14
Cu	26.00	28.78	24.96
Totals	100	100	100

Table 4: The average of (wt. %) of the detected elements in the studied metal objects before the conservation.

Element	A ring	a bracelet	a fishing pole
C	14.50	27.10	29.93
O	40.09	20.16	14.06
Na	-	-	-
Mg	1.49	0.63	0.31
Al	2.01	1.12	0.68
Si	5.99	2.85	1.76
S	0.49	-	0.16
Cl	4.31	8.56	11.46
K	0.57	0.29	0.25
Ca	8.48	0.81	0.18
Fe	1.42	0.46	0.29
Cu	20.65	38.03	39.57
Totals	100	100	100

3.2 Conservation processes

3.2.1 Conservation of the selected coins

The results of visual observation and microscopic examination showed that the coins, after mechanical cleaning, were covered with a thin layer of corrosion products and the surface morphology of the studied coins started to appear. However, the decoration and the writing symbols were not clear enough to be read on the coins. To show all decorations and writing symbols clearly, it was a necessity to complete cleaning but it was not able to clean them mechanically because continuing this process will destroy the coins. So, it was necessary to stop the mechanical cleaning process to prevent any scratch to the surface. For that it was decided to carry out other cleaning processes with chemical treatments.

The results of visual observation and microscopic examination of the coins after the chemical cleaning with Rochell salt solution showed that this process was effective in cleaning of the selected coins. The immersion of the coins in consecutive bathes of Rochell salt solution removed most of the corrosion products from the surface of the coins. After the cleaning with Rochell salt solution the results showed that one of the coin is a cupper coin and the second is silver coin. The results also showed that the coins became clean and most of corrosion products were removed from the surface of the coins. The writing and decoration on the surface of the coins appeared. For the silver coin it was recommended to follow this process by the treatment with Alkaline Dithionite Reduction Technique (Abdel-Kareem, et al, 2016). After the treatment of the silver coin with Alkaline Dithionite Reduction Technique, the coin became god and the writing and decoration on the surface of the coin became clear and can be read and studied with the archaeologists.

The results of SEM investigation of the coins after the chemical cleaning confirmed that the surface of the coins became smooth and the metal appeared well. The surface morphology of the cleaned coins showed that all corrosion products were removed. After the chemical cleaning, the SEM images of the coins showed an approximately homogenous surface composed from metal grains free from corrosion of large grains (see Figs. 8-9).

The results of the EDAX analysis for the surface of the coin 1 after the mechanical cleaning showed that the silver was appeared in the surface of the mechanical cleaned coin. The percent of the silver became about 13%; this indicated that this coin is silver coin (see table 5). The results of the EDAX analysis for the surface of the coin 1 after the mechanical cleaning indicated that the concentration of elements in the corrosion layer such as Cu, O, C, Al, Si, S, Cl, K, Ca and Fe became less than the same elements of the corrosion layer on the surface of the coins before cleaning (see table 1 and table 5). Although that the concentration of elements in the corrosion layer such as Cu, O, C, Al, Si, S, Cl, K, Ca and Fe were reduced, there was still a noticeable percent of these elements on the coin. These results confirmed that we did not reach the fresh surface of the coin. It was necessary to remove these contamination elements from the surface of the coin by using other cleaning methods. The presence of Cu along the surface of the coin after the mechanical cleaning gave us probability that it was added to the main component which is silver during the manufacturing of coin. Also, the presence of copper in high percentage in the surface of the corroded coin without cleaning indicated that the cu moved to the surface of the coin and chemical reaction between it and the surrounding environment occurred.

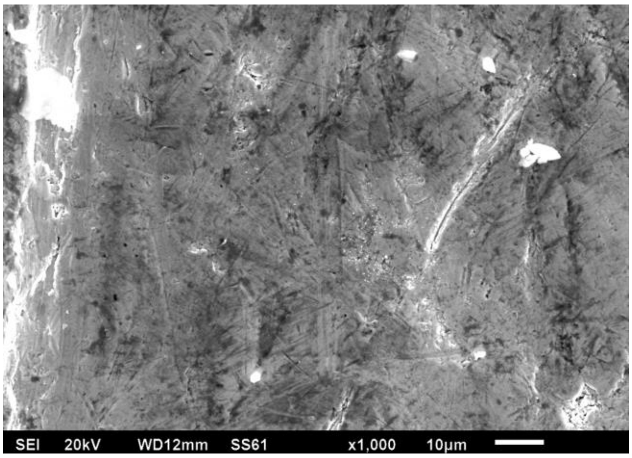

Figure 8 SEM image of the silver coin (coin 1) after the chemical cleaning, shows that the surface of the coin became smooth and appeared well.

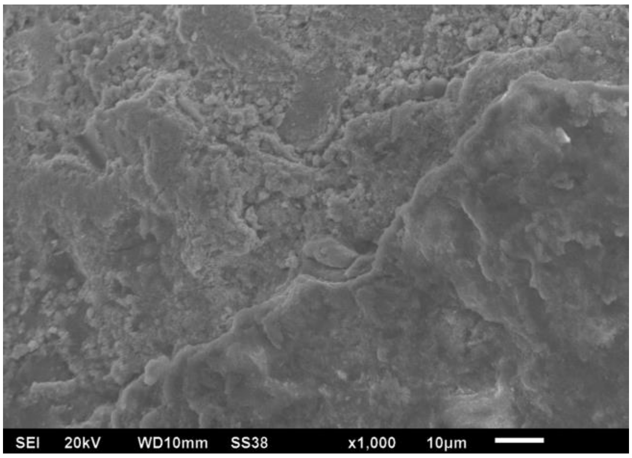

Figure 9 SEM image of the copper coin (coin 2) after the chemical cleaning, shows that the surface of the coin became smooth and appeared well.

The results of the EDAX analysis for the surface of the coin after treating with the alkaline Rochelle salt indicated that the main elements corrosion products such as Cu, O, C, Al, Si, S, Cl, K, Ca, and Fe were reduced or removed completely such as Al, Cl, K, and Fe. The main component of the fresh surface was silver (Ag) and copper (Cu) as a minor element. The comparison of EDX results before and after the treating with alkaline Rochelle salt revealed that the essential element of coin was the silver with concentration of 43%, while before the chemical cleaning (after the mechanical cleaning) was about 13%. This indicate that we became from the surface of the coin but there is a necessity to do more cleaning to appear the surface of the coin.

The results of the EDAX analysis for the surface of the coin after treating with Alkaline Dithionite Reduction Technique indicated that the main elements corrosion products such as O, C, Al, Si, S, Cl, K, Ca, and Fe were removed. The main component of the fresh surface was silver (Ag) and copper (Cu) as a minor element. The comparison of EDX results before and after the treating with Alkaline Dithionite Reduction Technique revealed that the essential element of coin was the silver with concentration of about 71%, while before the chemical cleaning (after the treating with alkaline Rochelle salt) was about 43%. This result indicate the coin became clean and ready to the study by archaeologists.

Table 5: The average of (wt. %) of the detected elements in the studied coin 1 while and the conservation

Element	Stage 1	Stage 2	Final
C	9.17	6.24	10.16
O	36.09	16.33	9.58
Na	-	-	-
Mg	2.84	0.93	-
Al	0.69	-	-
Si	2.79	0.84	-
S	0.29	3.06	-
Cl	0.67	-	-
K	0.42	-	-
Ca	9.04	1.19	-
Fe	0.36	-	-
Cu	24.42	27.80	9.41
Ag	13.22	43.62	70.58
Totals	100	100	100

The results of the EDAX analysis for the surface of the coin 2 after the surface cleaning showed that the copper percent became higher than before the surface cleaning. The percent of the copper (Cu) became about 54%. The results showed that there is no any traces of the silver (Ag). These results indicated that this coin is copper coin (see table 6). The presence of zinc (Zn) along the surface of the coin after the mechanical cleaning gave us probability that it was added to the main component which is copper (Cu) during the manufacturing of coin. These appearance of the zinc (Zn) on the surface of the coin 2 indicated that this coin may be made from the brass alloy. It is known that the brass alloy composed of copper and zinc. The results of the EDAX analysis for the surface of the coin 2 after the mechanical cleaning indicated that the concentration of elements in the corrosion layer such as O, C, Al, Si, S, Cl, K, Ca and Fe became less than the same elements of the corrosion layer on the surface of the coins before cleaning (see table 1 and table 6). Although that the concentration of elements in the corrosion layer such as O, C, Al, Si, S, Cl, K, Ca and Fe were reduced, there was still a noticeable percent of these elements on the coin. These results confirmed that we did not reach the fresh surface of the coin. It was necessary to remove these contamination elements from the surface of the coin by using other cleaning methods.

The results of the EDAX analysis for the surface of the coin after treating with the alkaline Rochelle salt in the first bath indicated that the main elements corrosion products such as O, C, Al, Si, S, Cl, K, Ca, and Fe were reduced. So it was necessary to repeat the treatment for many times. After the final treating with the alkaline Rochelle salt, the results of the EDAX analysis for the surface of the coin indicated that the main elements corrosion products such as O, C, Al, Si, S, Cl, K, Ca, and Fe were removed. The main component of the fresh surface was copper (Cu). The comparison of EDX results before and after the treating with alkaline Rochelle salt revealed that the essential element of coin was the copper with concentration of about 85%, while before the treating with alkaline Rochelle salt (after the mechanical cleaning) was about 54%. This result indicate the coin became clean and ready to the study by archaeologists.

Table 6: The average of (wt. %) of the detected elements in the studied coin 2 while and the conservation

Element	Stage 1	Stage 2	Final
C	9.54	11.67	11.22
O	19.06	12.87	3.37
Na	-	-	-
Mg	0.69	-	-
Al	1.59	0.31	-
Si	4.12	1.24	-
S	6.49	1.19	-
Cl	0.55	0.61	-
K	0.22	-	-
Ca	2.14	0.68	-
Fe	-	-	-
Cu	54.28	71.44	85.41
Zn	1.31		-
Totals	100	100	100

3.2.2 Conservation of the selected metal objects (rings, anklets, bracelets and fishing poles)

The results of visual observation and microscopic examination of metal objects (rings, anklets, bracelets and fishing poles) showed that the surface of metal objects, after mechanical cleaning, were still covered with a layer of corrosion products and the surface morphology of the studied metal objects started to appear. The results show that there is no decoration on the metal objects. As the surface of the metal objects appeared, so that it was necessary to stop the mechanical cleaning process to prevent any scratch to the surface. As it was necessary to appear the surface of the metal objects to be able to study with archaeologists, so that it was decided to carry out other cleaning processes with chemical treatments.

The results of visual observation and microscopic examination of the selected metal objects after the treating with Rochell salt solution showed that this process was effective in cleaning of the selected metal objects. The immersion of the metal objects in consecutive bathes of Rochell salt solution removed most of the corrosion products from the surface of the coins. After the treatment of the metal objects with Rochell salt solution, they became good and ready to be studied with the archaeologists (see Figs. 10-11).

Figure 10: Images of selected rings and bracelets, after the conservation processes.

Figure 11: Images of selected fishing poles after the conservation processes.

The results of SEM investigation of the selected metal objects (rings, anklets, bracelets and fishing poles) after the treating with Rochell salt solution confirmed that the surface of the metal became smooth and the metal appeared well. The surface morphology of the cleaned metal objects showed that the corrosion products were removed. After the treating with Rochell salt solution, the SEM photographing of the cleaned metal objects showed an approximately homogenous surface composed from metal grains free from corrosion of large grains (see Figs. 12-14).

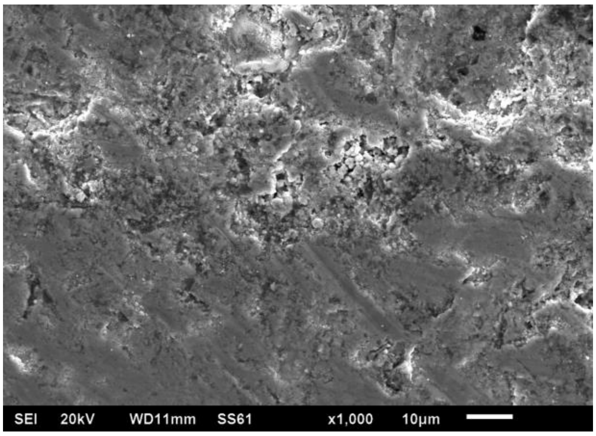

Figure 12 SEM image of the ring after the treating with Rochell salt solution, shows an approximately homogenous surface composed from metal grains free from corrosion of large grains.

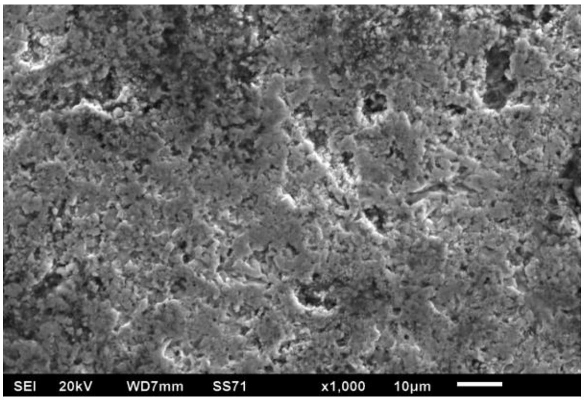

Figure 13 SEM image of the bracelet after the treating with Rochell salt solution, shows that the metal became smooth and the metal appeared well.

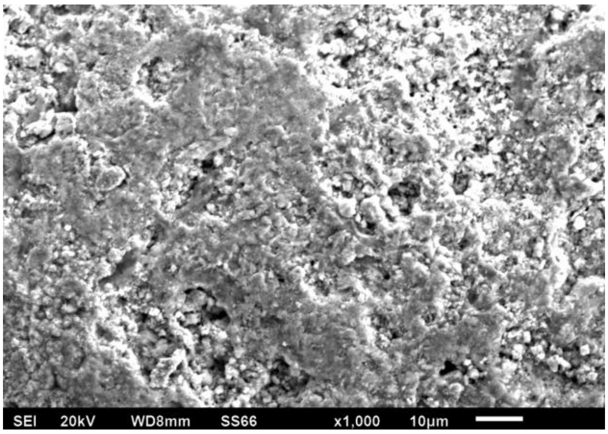

Figure 14 SEM image of the fishing pole after the treating with Rochell salt solution, shows that the metal became smooth and the metal appeared well.

The results of the EDAX analysis for the surface of the selected metal objects (rings, anklets, bracelets and fishing poles) after the cleaning processes showed that the copper percent became higher than before the cleaning (see table 7). The percent of the copper (Cu) became about 65 % in the ring, 63% in the bracelet, and 53% in the fishing pole. The presence of Tin (Sn) and lead (Pb) along the surface of the bracelets and fishing poles after the cleaning gave us probability that they were added to the main component which is copper (Cu) during the manufacturing of anklets, bracelets and fishing poles. These appearance of tin (Sn) and lead (Pb) on the surface of the conserved metals indicated that the anklets, bracelets and fishing poles may be made from the bronze alloy. It is known that the bronze alloy composed of copper (Cu), tin (Sn) and lead (Pb).

The results revealed that the substrate of rings is copper that contains a high percent of Cu (64.57%), turned to Cuprite in most parts where O is the minor element (14.02%). The results showed that there is no any traces of tin (Sn) and lead (Pb) on rings. These results indicated that the rings are made from copper. However these results did not confirm that the rings may not be made from bronze alloy or other copper alloy. As it was confirmed in previous studies that tin (Sn) and lead (Pb) while their burial time move to the surface of the metal objects, so that these elements are much more easily leached out than the main metal (copper) due to the chemical reaction between them and the surrounding environment occurred (Ghoniem, 2011) .

Table 7: The average of (wt. %) of the detected elements in the studied metal objects after the conservation.

Element	A ring	bracelet	fishing pole
C	11.70	9.53	11.64
O	14.02	13.28	20.75
Na	-	-	-
Mg	0.55	-	1.20
Al	0.56	-	-
Si	1.00	-	0.35
S	-	2.07	-
Cl	0.71	8.31	0.48
K	-	-	-
Ca	0.90	-	-
Fe	-	0.32	-
Cu	64.57	62.94	52.69
As	-	-	0.01
Sn	-	2.44	6.60
Pb	-	1.11	4.21

Totals	100	100	100

The results revealed that the substrate of bracelets is bronze that contains a high percent of Cu (62.94%), turned to Cuprite in most parts where O is the minor element (13.28%), together with Pb (1.11%) and Sn (2.44%).

The results revealed that the substrate of fishing poles is bronze that contains a high percent of Cu (52.69%), turned to Cuprite in most parts where O is the minor element (20.75%), together with Pb (4.21%) and Sn (6.60%).

The results of the EDAX analysis for the surface of the selected metal objects (rings, anklets, bracelets and fishing poles) after the cleaning processes that the main elements corrosion products such as O, C, Al, Si, S, Cl, K, Ca, and Fe were reduced. The obtained results are enough so it was not necessary to carry out more cleaning for them. These results indicated that the selected metal objects (rings, anklets, bracelets and fishing poles) became clean and ready to the study by archaeologists.

4. ARCHAEOLOGICAL AND HISTORICAL STUDIES FOR THE TREATED METAL OBJECTS

For authentication of these selected metal objects, it was important to read all decorations, inscriptions, and writings on the surface of the conserved coins. In relevance to the Islamic coins, rulers, emphasize more on the words instead of images. Because of this, the messages communicated through the Islamic coins, they are longer and more explicit (AlJaber, 2015). Imitation of nature does not take an important place in Islamic culture so has not been widely used in Islamic coins (Salehi, et al, 2015).

After the final treatment of the coins, different inscriptions and writing words appeared on the surfaces. The coin 1 is an Islamic dirham (DH) made from the silver (Ag) (see Figs. 15-16). While the coin 2 is an Islamic penny made from the copper (Cu) (see Figs. 17-18). The drawings and inscriptions found on the coins confirm that these coins may be Ayyubid coinages that belong to Ayyubid dynasty that date back from 569 to 649 H.D. (1147-1252 A.D.).

The Ayyubid dynasty was a Muslim dynasty of Kurdish origin founded by Saladin and centered in Egypt. The dynasty ruled large parts of the Middle East during the 12th and 13th centuries (see Fig. 19). The Ayyubid Sultanate included Egypt, Syria, Upper Mesopotamia, the Hejaz, Yemen and the North African (Ayyubid dynasty, 2019).

If we examine the name of the king on the silver dirham (coin 1) as well as his ruling time, we will find that the coin 1 date back to (1242 – 1258 A.D.) (640- 656 H.). This coin date back to the end of the Ayabe Period. It is a Dirham (DH) Ayabe, it was written on the obverse of it in the center of this coin, in Arabic words, texts shows Al-Imam, Al-Musta'sim Billah, Abu Ahmed, Abd Allah, prince of the faithful. The year of beating of this coin, is unclear (see Fig. 17). It was written on the obverse of this coin, in Arabic words, texts show the King (Al-Mansur Abu Bakr) And on the right side of the margin, Arabic text show No God except Allah (see Fig. 18).

It is known that Al-Musta'sim Billah, was Last Abbasid Caliph in Baghdad; executed after the Mongol sack of Baghdad. Al-Musta'sim Billah, was the 37th and last Caliph of the Abbasid Caliphate; he ruled from 1242 A.D. until his death. The presence of the name of the Abbasid Caliphate, Al-Musta'sim Billah Abu Ahmed, on the observe of the coin and the name of Ayyubid Sultan (Al-Mansur Abu Bakr) on the reverse confirms that the Ayyubid Sultans in this time were follow in their roller to Abbasid Caliphate in Baghdad.

Figure 15: Image of the obverse of the coin 1 after the conservation processes.

Figure 16: Image of the reverse of the coin 1 after the conservation processes.

If we examine the name of the king on the copper penny (coin 2) as well as his ruling time, we will find that this penny may be Ayyubid coin. It was written on the obverse of it in the center of this coin, in Arabic words, texts shows the Abbasid Caliphate (Al-Nasir li-Din Allah Abu Alabbas), prince of the faithful. The year of beating of this coin, is unclear (see Fig. 17). We could not read the text on the reverse of the coin (see Fig. 18). The presence of the name of the Abbasid Caliphate (Al-Nasir li-Din Allah Abu Al-abbas), on observe of the copper penny, confirms that this penny, is date back to 575 to 622 H.D. (1180 – 1225 A.D.).

Figure 17: Image of the obverse of the coin 2 after the conservation processes.

Figure 18: Image of the reverse of the coin 2 after the conservation processes.

Figure 19: Map of Ayyubid Sultanate (in green color) shows the Ayyubid dynasty at its greatest extent, founded by Saladin 1188 A.D.

5. CONCLUSION

The best method that can be used safely and successfully for conservation of archaeological silver coins excavated from Al-Serein excavation is as the following. In the beginning the corroded coins have to be treated with alkaline Rochelle salt. Then they should be treated with alkaline dithionite reduction technique. Following the final cleaning, the coins have to be rinsed with de-ionized water. After the rinsing process, the coins have to be dried well. Finally after finishing the treatments, the coins have to be isolated and coated with a clear acrylic lacquer such as Palaroid B44.

The best method that can be used safely and successfully for conservation of archaeological cupper metal objects (coins, rings, bracelets and fishing poles), excavated from Al-Serein excavation is as the following. In the beginning the corroded metal objects have to be cleaned with alkaline Rochelle salt. After the final cleaning, the cupper metal objects have to be rinsed with de-ionized water. After the rinsing process, the metal objects have to be dried well. Then the metal objects have to degrease with acetone, and follow that with 3% on Benzotriazole in ethanol. Finally after finishing the treatments, the metal objects have to be isolated and coated with a clear acrylic lacquer such as Palaroid B44.

The archaeological study of the decorations and inscriptions that appeared on the surface of the coins after their conservation confirms that these coins are Ayyubid coins which date back to Ayyubid dynasty 569 to 649 H.D. (1147-1252A.D.). They are include silver dirham and cupper penny. The presence of the name of the Abbasid Caliphate (Al-Musta'sim Billah Abu Ahmed), on the observe of the silver dirham and the name of Ayybid king (Al-Mansur Abu Bakr) on the reverse confirm that this dirham is date back to 640 to 656 H.D. (1242 – 1258 A.D.). The presence of the name of the Abbasid Caliphate (Al-Nasir li-Din

Allah Abu Alabbas), on observe of the copper penny, confirms that this penny, is date back to 575 to 622 H.D. (1180 – 1225 A.D.).

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