

# The corrosion characteristics and electrochemical conservation treatment for an archaeological lead ossuary from Jordan

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**Abstract:** This study investigates the composition and corrosion of a lead ossuary excavated from the archaeological cemetery located on the site of the Queen Alia International Airport, Jordan. It also presents its conservation treatment using the potentiostatic reduction method. X-ray fluorescence (XRF) and scanning electron microscopy/ energy dispersive X-ray spectroscopy (SEM/EDX) analyses showed that the ossuary was made from a relatively pure lead metal. Examination of the corrosion products using X-ray diffraction (XRD) and SEM/EDX analyses indicated the presence oxides, carbonates and chlorides of lead as the main corrosion products on the ossuary. The metallographic examination showed a cast  $\alpha$ -Pb dendritic microstructure. A linear sweep voltammetry (LSV) scan for a small fragment from the ossuary in a sodium sulfate solution allowed identifying the working conditions to reduce the corrosion products to lead metal. Chronoamperometry (CA) techniques helped to monitor the reduction process and determine its completion. The potentiostatic methods showed an excellent effectiveness in determining treatment parameters and monitoring the reduction process of the corrosion products on the ossuary and to prevent over-treatment. The reduced ossuary was finally coated with Paraloid B72 to prevent corrosion reoccurrence in the poor environmental conditions of the storage area.

**Keywords:** Lead, ossuary, corrosion, analysis, conservation, potentiostatic reduction

## Las características de corrosión y el tratamiento de conservación electroquímica de un osario de plomo arqueológico de Jordania

**Resumen:** Este estudio investiga la composición y corrosión de un osario de plomo excavado en el cementerio arqueológico ubicado en el sitio del Aeropuerto Internacional Queen Alia, Jordania. También se presenta su tratamiento de conservación mediante el método de reducción potenciostática. Los análisis de fluorescencia de rayos X (XRF) y microscopía electrónica de barrido/espectroscopía de rayos X de dispersión de energía (SEM/EDX) mostraron que el osario estaba hecho de un metal de plomo relativamente puro. El examen de los productos de corrosión mediante análisis de difracción de rayos X (XRD) y SEM/EDX indicó la presencia de óxidos, carbonatos y cloruros de plomo como los principales productos de corrosión en el osario. El examen metalográfico mostró una microestructura dendrítica colada de  $\alpha$ -Pb. Un escaneo de voltamperometría de barrido lineal (LSV) para un pequeño fragmento del osario en una solución de sulfato de sodio permitió identificar las condiciones de trabajo para reducir los productos de corrosión a plomo metal. Las técnicas de cronoamperometría (CA) ayudaron a monitorear el proceso de reducción y determinar su finalización. Los métodos potenciostáticos mostraron una excelente efectividad para determinar los parámetros de tratamiento y monitorear el proceso de reducción de los productos de corrosión en el osario y para prevenir el sobretreatment. El osario reducido finalmente se recubrió con Paraloid B72 para evitar la recurrencia de la corrosión en las malas condiciones ambientales del área de almacenamiento.

**Palabras clave:** Plomo, osario, corrosión, análisis, conservación, reducción potenciostática

## As características de corrosão e tratamento de conservação eletroquímica para um ossuário de chumbo arqueológico da Jordânia

**Resumo:** Este estudo investiga a composição e corrosão de um ossuário de chumbo excavado no cemitério arqueológico localizado no local do Aeroporto Internacional Queen Alia, na Jordânia. Apresenta também seu tratamento de conservação pelo método de redução potenciostática. As análises de fluorescência de raios X (XRF) e microscopia eletrônica de varredura/espectroscopia de energia dispersiva de raios X (SEM/EDX) mostraram que o ossuário era feito de um metal de chumbo relativamente puro. O exame dos produtos de corrosão

por difração de raios X (DRX) e análises SEM/EDX indicou a presença de óxidos, carbonatos e cloretos de chumbo como os principais produtos de corrosão no ossuário. O exame metalográfico mostrou uma microestrutura dendrítica  $\alpha$ -Pb fundida. Uma varredura de voltametria de varredura linear (LSV) para um pequeno fragmento do ossuário em uma solução de sulfato de sódio permitiu identificar as condições de trabalho para reduzir os produtos de corrosão a chumbo metálico. As técnicas de cronoamperometria (CA) ajudaram a monitorar o processo de redução e determinar sua conclusão. Os métodos potenciostáticos mostraram uma excelente eficácia na determinação dos parâmetros de tratamento e monitoramento do processo de redução dos produtos de corrosão no ossuário e na prevenção do tratamento excessivo. O ossuário reduzido foi finalmente revestido com Paraloid B72 para evitar a recorrência de corrosão nas más condições ambientais da área de armazenamento.

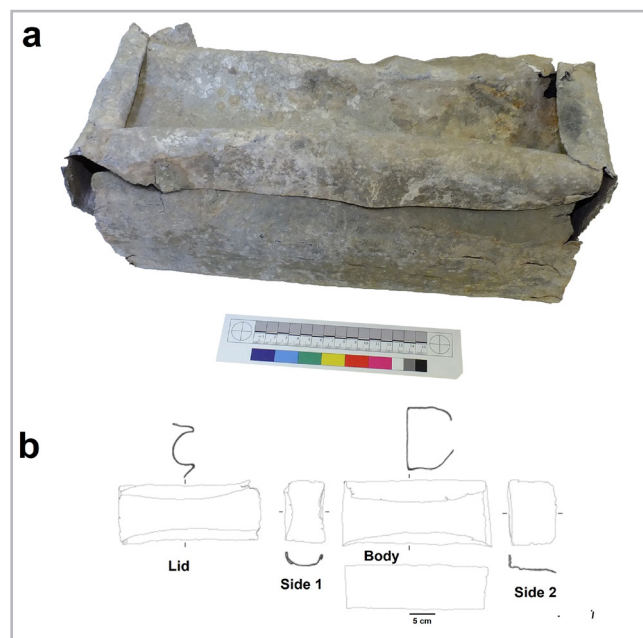
**Palavras-chave:** Chumbo, ossuário, corrosão, análise, conservação, redução potenciostática

## Introduction

Lead is one of the earliest known and used metals. Its abundance, ease of extraction from its ores, low melting point, softness, flexibility, and relative corrosion resistance made it suitable to produce useful objects and architectural elements (Craddock 1995: 205; Selwyn 2004: 115). The archaeological lead and lead alloy artifacts form an important part of the cultural heritage collections. Therefore, their conservation is a main interest for archaeological metal conservators and conservation scientist.

The common lead corrosion products are stable, however they mostly form a white extended corrosion layer that obscures the bluish-grey original lead surface, therefore distorts the identifying visual characteristics of the object, and hides its surface details. In the professional conservation practice, the common interventive approaches used to conserve archaeological lead objects include mechanical, chemical, and electrochemical methods (Costa and Urban 2005, Schotte and Adriaens 2006, Palomar and Cano 2018). Since the hardness of lead is lower than the hardness of its common corrosion products, the mechanical cleaning of those corrosion products makes the underlying lead metal susceptible to scratching and damage (Cronyn 1990, 205–207). On the other hand, the chemical cleaning by acids, bases or chelating agents can remove the corrosion products from the surface, however they can also attack the metal, which is a main disadvantage for objects with non-uniform corrosion, or if remains of the solutions were not cleaned well after the treatment (Caley 1955; Watson 1985; Švadlena *et al.* 2020). The electrochemical reduction methods, particularly the potentiostatic reduction methods, have the advantage of consolidating the lead objects and minimizing the mass loss by returning the corrosion products back to metallic lead. The reduction of the corrosion products on a fixed sufficient potential while avoiding hydrogen evolution preserves the surface details that characterize the object, and the reduced metallic form clarifies the original identity of the object without obliterating its antique appearance and cultural values. The treatment is effective for lead objects with stable corrosion products or active corrosion products promoted by the presence of organic acid attacking the object (Carradice and Campbell 1994; Degriigny and Le Gall 1999; Rocca *et al.* 2004; Schotte *et al.* 2006; Barrio *et al.* 2009; Fontaine *et al.* 2016; Palomar *et al.* 2018).

This study investigated the corrosion of a lead ossuary from the collection of the Museum of Jordanian Heritage at Yarmouk University, Irbid, Jordan [Figure 1]. The ossuary came originally from a rescue excavation that was conducted at a late 2<sup>nd</sup> – early 3<sup>rd</sup> AD century cemetery site, which was discovered during the construction works of the Queen Alia International Airport, Amman back in 1978 (Ibrahim and Gordon 1987). The ossuary is relatively corroded, and no previous analyses or conservation treatments have been conducted on it since its acquisition and placement in the storage area of the museum. The study analytically investigated the composition of the original material and corrosion products of the ossuary and applied a conservation treatment using the potentiostatic reduction method. Finally, the ossuary was coated with Paraloid B-72 protective coating to prevent corrosion reoccurrence in the non-ideal storage environment.



**Figure 1.-** An image (a) and drawing (b) illustrating the lead ossuary before the treatment

## Materials and methods

The mineralogical composition of the corrosion products on the lead ossuary was determined using a Shimadzu LabX, XRD-6000 X-ray diffractometer. Copper  $K\alpha_1$  radiation

( $\lambda=1.5418\text{\AA}$ ) was used for the analysis and the spectrum was acquired at 40 kV, 30 mA and a step size of  $0.05^\circ$  in  $2\theta$ . The elemental compositions of the ossuary and the solder used to join its plates were determined by X-ray fluorescence (XRF) analysis. A S1 Titan 600 hand-held XRF analyzer (Bruker, Germany) equipped with a high-performance Silicon Drift Detector (SDD) was used in the analysis. The XRF analysis was performed at 40 kV excitation, 5 mm spot size and 60 s acquisition time. The small analysis sites on the of the body and solder point were first cleaned and flattened with a dental microdrill to reach an uncorroded surface. The quantitative analysis results were acquired with the aid of an internal alloy grade library. SEM/EDX was also used to investigate the elemental composition of the corrosion products and original material of the ossuary. Two cross-sections were taken from the body of the ossuary using a fine jeweller's saw and embedded in an Araldite 2020 epoxy resin. Following curing for 24 hours, a cross-section was ground on a series of silicon carbide papers from 1000 to 3000 grit and then polished on a Buehler minimet polisher beginning at 9–3  $\mu\text{m}$  alumina polishing suspensions and reducing down to a highly polished surface using 1–0.25  $\mu\text{m}$  diamond pastes. The sample was then decontaminated in ethanol in an ultrasonic bath and finally dried. The SEM/EDX investigation was carried out using a scanning electron microscopy (SEM) coupled with an energy dispersive spectroscopy (EDX) analyzer (FEI-Quanta 200). The second embedded cross-section was only polished with 9–3  $\mu\text{m}$  alumina polishing suspensions then decontaminated in ethanol in an ultrasonic bath and dried. This cross-section was etched with the acidified glycerol etchant (Scott 1992: 71), then it was investigated using an Optika IM-3MET inverted metallurgical microscope. A cross-polarized image of the corrosion profile and internal microstructure was taken using an Optika C-P8 digital camera attachment.

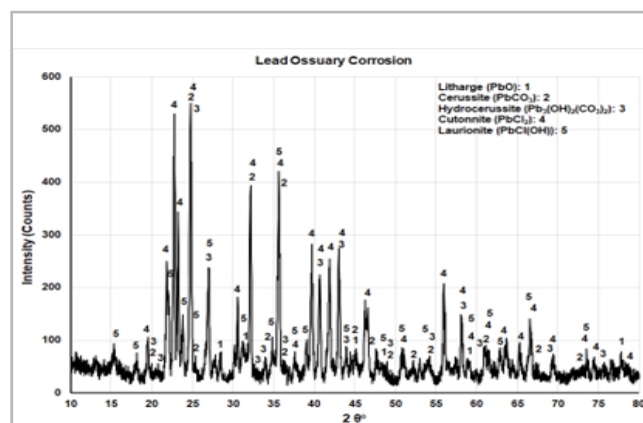
A small broken fragment (2 x 1 x 0.1 cm) from the ossuary was used for determining the potentiostatic reduction parameters of the corrosion products. A three-electrode electrochemical cell connected to a potentiostat (Vertex<sup>®</sup>, Ivium Technologies, The Netherlands) was prepared, the corroded lead fragment served as the working electrode of the cell and connected to the alligator clip of the working electrode's wire outside the electrolyte solution by a platinum needle that was inserted in it, mercury/ mercurous sulfate ( $\text{Hg}/\text{Hg}_2\text{SO}_4$ ) electrode filled with saturated potassium sulfate (10% w/w) (Gamry Instruments) served as the reference electrode, a T-316 stainless-steel mesh with a surface area bigger than the surface area of the corroded lead fragment served as the counter electrode, and a 100 ml of 0.1 M sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) solution (pH= 7) was used as the electrolytes. Linear sweep voltammetry (LSV) in the cathodic direction was carried out from the corrosion potential ( $E_{\text{corr}}$ ) to  $-1.4$  V vs the  $\text{Hg}/\text{Hg}_2\text{SO}_4$  reference electrode with a scanning rate of 1.0 mV/s. The potential of the reference electrode is 0.615 V vs the normal hydrogen electrode (NHE). Before the LSV experiment, the corroded lead fragment was

decreased in acetone to ensure complete wetting of the metal surface. Based on the LSV scan result of the small fragment, the ossuary's plates were individually treated by potentiostatic reduction processes carried out at  $-1.3$  V in the same electrolyte (0.1 M  $\text{Na}_2\text{SO}_4$ ). This potential was sufficient to assure a complete reduction of all corrosion products while avoiding hydrogen evolution that may obliterate the reduced metal surface. A T-316 stainless-steel mesh with a surface area bigger than the surface area of each corroded lead plate was used as the counter electrode of the cell. The volume of the electrolyte was 2 L for each of the two small sides of the ossuary, 8 L for the lid and 16 L for the main body. The approximate surface areas of the ossuary's plates were the following: 580  $\text{cm}^2$  for each of the two small sides, 2300  $\text{cm}^2$  for the lid, and 4100  $\text{cm}^2$  for the body. The reduction processes were monitored by chronoamperometry (CA) to determine the end-point of the treatment, which is achieved by having a low and stable current value indicating the completion of the reduction process (Costa 2003; Fontaine *et al.* 2016; Abu-Baker and MacLeod 2021). A small cross-section sample was taken from the ossuary after the potentiostatic reduction treatment. It was embedded and prepared as described for the sample before the treatment, then examined using the optical microscope to investigate the change on the corrosion layer. The ossuary was finally coated with a 5% Paraloid B72 dissolved in acetone and applied by brushing for 3 times to form a protective layer on its freshly reduced and porous metallic surface.

## Results and discussion

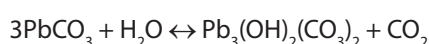
### —XRD analysis

The XRD characterization of the corrosion products on the lead ossuary is shown in Figure 2. The diffractogram shows that the lead (II) chloride (cotunnite  $\text{PbCl}_2$ ) is the main corrosion product with the presence of lead (II) oxide (litharge  $\text{PbO}$ ), lead (II) carbonate (cerussite  $\text{PbCO}_3$ ), basic lead (II) carbonate (hydrocerussite  $\text{Pb}_3(\text{OH})_3(\text{CO}_3)_2$ ), and lead (II) chloride hydroxides (laurionite  $\text{PbCl}(\text{OH})$ ).



**Figure 2.-** XRD analysis result of the corrosion products on the lead ossuary

Lead is generally resistant to corrosion; it forms a thin layer of lead oxide (litharge  $\alpha$ -PbO) protective layer when it corrodes in the atmosphere. Lead carbonates are expected corrosion products for artifacts excavated from carbonate containing soils. Over time, the lead oxide reacts with carbonate ions and forms carbonate compounds, mainly lead carbonate (cerussite  $\text{PbCO}_3$ ) and basic lead carbonate (hydrocerussite  $\text{Pb}_3(\text{OH})_3(\text{CO}_3)_2$ ). These carbonate compounds eventually form an adherent, relatively insoluble layer that decreases the corrosion rate of the lead. Cerussite ( $\text{PbCO}_3$ ) and hydrocerussite ( $\text{Pb}_3(\text{OH})_3(\text{CO}_3)_2$ ) are in equilibrium at a carbon dioxide pressure of  $10^{-2.8}$  atm as the following equation shows:



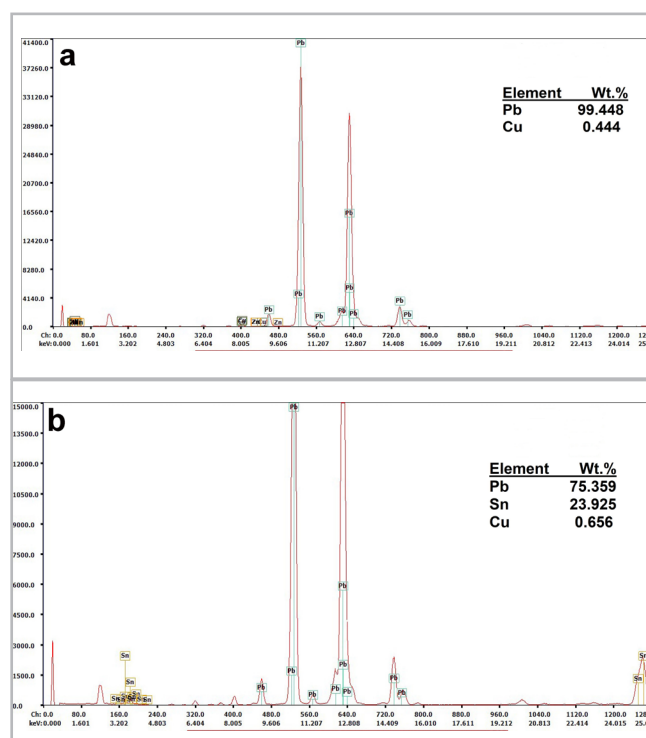
Cerussite is the main corrosion product of lead artifacts buried in aerobic alkaline carbonate soil, where the carbon dioxide pressure is usually about  $10^{-2}$  atm, which is above the equilibrium value, while hydrocerussite ( $\text{Pb}_3(\text{OH})_3(\text{CO}_3)_2$ ) is the main corrosion product of lead artifacts exposed to the open atmosphere for a long time, because the atmospheric pressure of carbon dioxide (i.e.  $10^{-3.5}$  atm) is less than the equilibrium value (Turgoose, 1985). The ossuary had been excavated from a carbonate soil then stored in an open atmosphere, which could justify the presence of the two corrosion products on its surface (Mattias *et al.* 1984; Nosek 1985; Abu-Baker *et al.* 2014).

The presence of the lead chloride-containing compounds (cotunnite  $\text{PbCl}_2$  and laurionite  $\text{PbCl}(\text{OH})$ ) as corrosion products indicate the presence of dissolved chloride anions in the burial soil of the ossuary (Selwyn 2004: 122). The percolation of saline water to the burial soil causes the formation of lead chloride and oxychloride minerals, which can also associate with the present carbonate ions to form double salts under certain conditions. Cotunnite ( $\text{PbCl}_2$ ) is stable under acidic environments and high chloride concentrations. On the other hand, laurionite  $\text{PbCl}(\text{OH})$  is formed when the pH of natural saline environment (i.e. chloride concentration is about 0.5 M and pH is about 8) is increased, penfieldite  $\text{Pb}_2(\text{OH})_3\text{Cl}$  would also form when the pH is increased but it is thermodynamically less stable than laurionite. Therefore, the presence of cotunnite  $\text{PbCl}_2$  as a main corrosion product suggests high chloride concentrations in the burial soil, and the mixture of corrosion products could be attributed to various environmental conditions that affected the object in the burial context and storage area (Abdul-Samad *et al.* 1982; Turgoose 1985, Edwards *et al.* 1992).

#### — XRF

The XRF analysis results of the ossuary's body and solder are shown in Figure 3. The ossuary is made of a relatively pure lead metal, while the solder used to join the now separated short sides to the main body of the ossuary is made of a lead (Pb)-tin (Sn) alloy, which contains about 24% Sn, therefore

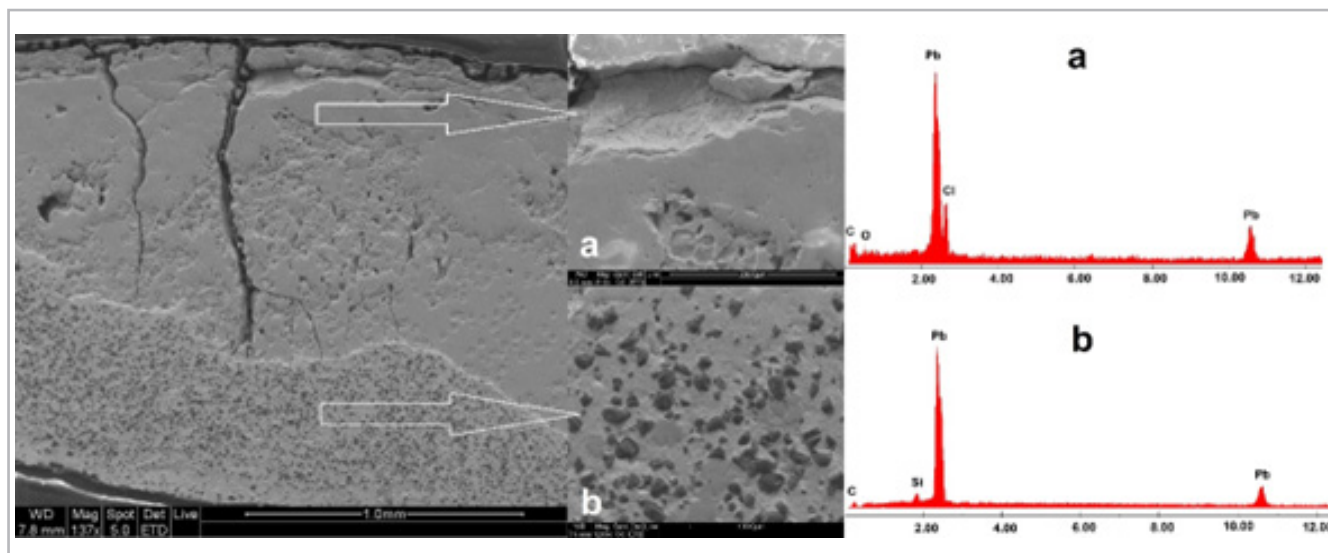
had a lower melting point than the pure lead used to make the ossuary. The plates used for the manufacture of the ossuary would have been prepared by the sand-casting technique. To make the ossuary, a single rectangular sheet of lead was bent up at the side to form the main part of the coffer. The small side plates were soldered to the main part with a Pb-Sn alloy so as to complete the rectangular coffer. The lid of the ossuary overlaps the coffer and was made using the same casting and bending procedures. (Gowland 1901; Rocca *et al.* 2004; Prabhakar *et al.* 2019).



**Figure 3.-** XRF spectra and quantitative elemental analysis results for the ossuary (a) and its solder (b)

#### — SEM/EDX analysis and optical microscopy

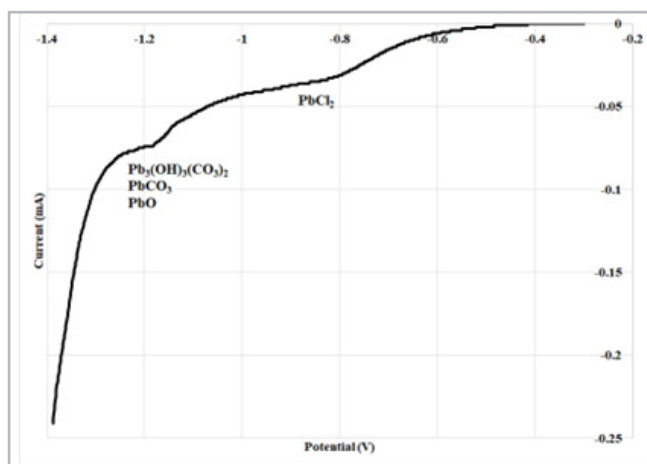
The SEM/EDX examination of the corrosion profile of a cross section taken from the body of the lead ossuary shows a cracked irregular corrosion layer with a thickness that reaches about 1000 microns in some areas above the internal uncorroded metal [Figure 4]. The EDX analysis shows that the corrosion layer (area-a in the SEM image) mainly contains lead, oxygen, carbon, and chlorine. This suggests the presence of oxides, carbonate and chloride corrosion products of lead, which conforms to the XRD analysis results. The EDX analysis of the grey internal uncorroded metal (area-b in the SEM image) shows that the ossuary was cast from a relatively pure lead metal without adding alloying metals. The presence of carbon and silicon in the EDX analysis is expected because of the penetration of abrasive materials into the soft lead surface during the polishing of the cross-sections (Scott 1996). The silicon carbide abrasive residues appear as black particles in the SEM image.



**Figure 4.-** SEM images and EDX analysis for the corrosion products (a) and internal uncorroded metal of the ossuary (b)

#### — LSV scan

Determining the reduction potentials for the lead corrosion productions on the ossuary was carried out by linear sweep voltammetry (LSV) in 0.1 M  $\text{Na}_2\text{SO}_4$ . Figure 5 shows the LSV plot of the cathodic polarization of the small broken fragment of the ossuary in 0.1 M  $\text{Na}_2\text{SO}_4$ . The plot shows two broad reduction peaks; one that reaches its maximum current at about -0.9 V and can be attributed to the reduction of the cottunite ( $\text{PbCl}_2$ ) corrosion product. The following broad reduction peak reaches its maximum current at about -1.2 V and can be attributed to the reduction of the litharge ( $\text{PbO}$ ), cerussite ( $\text{PbCO}_3$ ) and hydrocerussite ( $\text{Pb}_3(\text{OH})_2(\text{CO}_3)_2$ ) corrosion products present on the surface as indicated by the XRD and SEM/EDX analyses. The hydrogen evolution potential starts after -1.3 V (Degryny and Le Gall 1999; Selwyn 2017). Since ossuary had a mixture of corrosion products on the surface, their close reduction peaks overlapped and formed a single broad reduction peak. However, the SEM/EDX and XRD aided the interpretation of the LSV plot.

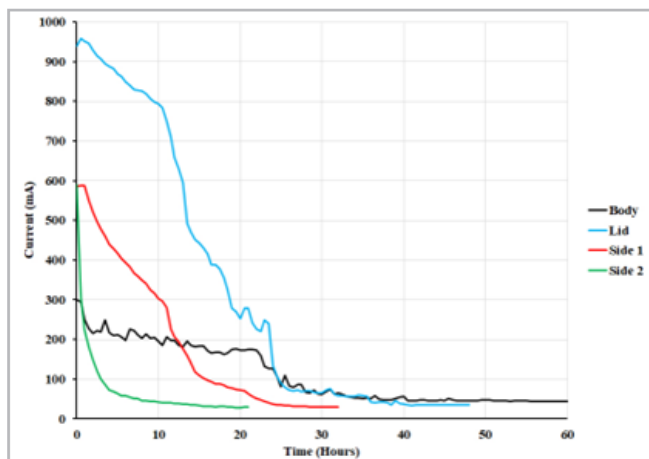


**Figure 5.-** Linear sweep voltammetry plot for the small corroded fragment taken from the lead ossuary

#### — Potentiostatic reduction

The lead ossuary has four separate plates: the body, lid and two sides. Each part was cathodically polarized at a constant potential of -1.3 V to assure a complete reduction of all corrosion products and avoid vigorous hydrogen evolution. Each of the four ossuary's parts was treated separately, and the process was monitored by chronoamperometry. Monitoring the current of side 1 plate showed that it started from about 585 mA at the beginning of the potentiostatic reduction process and decreases to about a stable low value of 29 mA after 32 h of potentiostatic reduction at -1.3 V. For the side 2 plate, the current started at 589 mA and reached about a stable value of 28 mA after 21 h of the reduction process. For the lid of the ossuary, the current decreased from about 939 mA to a low stable value of about 35 mA indicating the completion of the reduction process after 48 h. Finally, the reduction current of the body of the ossuary decreased from 300 mA to 38 mA in about 60 h of the potentiostatic reduction process [Figure 6]. The progressive decrease in the current indicates a progressive reduction of the corrosion products. At the end of the reduction process, the pH of the electrolyte decreased to about 3, which indicates that the production of  $\text{H}^+$  ions at the anode was not compensated by similar production of  $\text{OH}^-$  ions at the cathode, where the main reaction process was the reduction of the lead corrosion products (Degryny and Le Gall 1999).

The potentiostatic reduction treatment converted the corrosion products on the ossuary to a reduced porous grey metal deposited on the surface. Therefore, after the completion of reduction process, each part of the ossuary was rinsed under running water with gentle brushing to clean the residual encrustations and improve its appearance, then it was cleaned with deionized water, followed by drying in acetone.

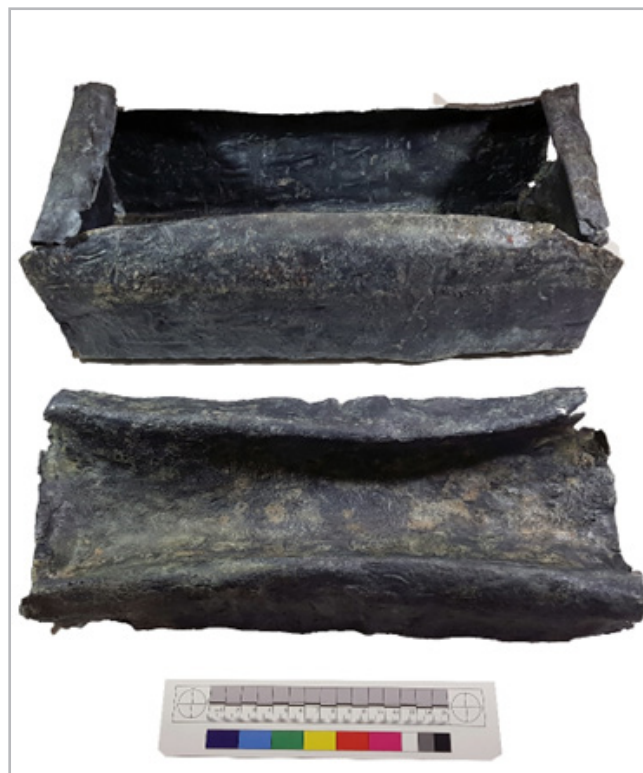


**Figure 6.-** Chronoamperometric curves for the potentiostatic reduction of the corrosion products on the four parts of the lead ossuary

— *Optical microscopy and protective coating*

The polarized light microscopy image of the cross section taken from the lead ossuary before the treatment shows a white corrosion layer over the internal grey lead metal. The image shows a cast  $\alpha$ -Pb dendritic microstructure. The composition of the corrosion layer was identified by the XRD and SEM/EDX analysis. The hardly appearing dendrites of the uncorroded lead are distorted and have oriented patterns, which are associated with the direction of heat extraction and cooling rate of the cast sheet [Figure 7.a] (Vander Voort 2004; Costa and Urban 2005). The microscopic image of the sample taken from the ossuary after the potentiostatic reduction treatment shows a grey porous over the internal sound metal [Figure 7.b]. This indicates the success of the potentiostatic reduction process in converting all corrosion products into reduced powdery metallic lead over the internal sound lead metal (Degryny and Le Gall 1999).

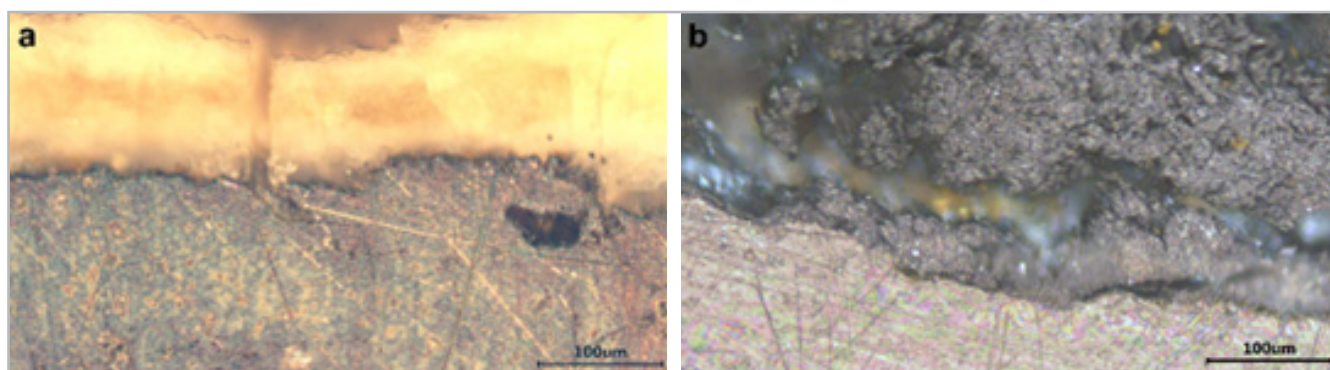
Given that the ossuary was going to a non-ideal storage environment, it was coated with a 5% Paraloid B72 applied by brushing for 3 times to form a protective coating layer on its surface. Figure 8 shows the images of the artifacts after the end of the treatment.



**Figure 8.-** The ossuary after the treatment

**Conclusion**

The spectroscopic and microscopic analytical investigation of the lead ossuary from the archaeological cemetery located at the Queen Ali International Airport site identified its original composition, corrosion products, and state of preservation. The linear sweep voltammetry plot of a sample from the ossuary helped to identify the required working parameters to reduce the corrosion products while avoiding over potential causing vigorous hydrogen evolution, and the chronoamperometry technique was effective in determining the end of the process. The potentiostatic reduction method was effective in conserving the ossuary by returning its corrosion products back to a metallic form that clarifies its material nature without obliterating its antique appearance and cultural values.



**Figure 7.-** Polarized light microscopy image of the corroded edge and internal metal of the ossuary (a), and microscopic image of a cross section taken from the ossuary after the potentiostatic reduction process (b)

## Acknowledgements

I would like to thank the Deanship of Scientific Research and Graduate Studies at Yarmouk University for funding this research. I would also like to thank Professor Moawiya Ibrahim for kindly offering me a written permission to work on the ossuary and publish the results. I also thank Sereen Al-Shoubaki for drawing the sketch illustrating the four parts of the ossuary.

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Artículo enviado 07/02/2022  
Artículo aceptado el 28/11/2022



<https://doi.org/10.37558/gec.v22i1.1093>