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Treatment and Protection of a Historic Brass Candelabra from the French Institute of Oriental Archaeology Using Nano-TiO_2 Enhanced Benzotriazole

Mohamed Sedek

Conservation Department, Faculty of Archaeology, Fayoum University, Fayoum, Egypt; Conservation Department, Faculty of Archaeology and Tourism Guidance, Misr University for Science and Technology.
mohamed.sedek@must.edu.eg

Ragab Abou EL-Hassan

Conservation Department, Faculty of Archaeology and Tourism Guidance, Misr University for Science and Technology, Egyp
raga.sayed@must.edu.eg

Saleh M. Saleh

Conservation Department, Faculty of Archaeology, Fayoum University, Fayoum, Egypt.
sms02@fayoum.edu.eg

Mohamed Megahed

Conservation Department, Faculty of Archaeology, Fayoum University, Fayoum, Egypt.
mmm03@fayoum.edu.eg

ARTICLE INFO

Article history
Received 9 April 2025
Received in revised form 14 May 2025
Accepted 17 May 2025
Available Online 17 July 2025

KEYWORDS

Brass Alloy, Copper-Zinc, Historic Candelabra, Benzotriazole (BTA), Nanoparticles (TiO_2), Corrosion inhibition, Conservation

ABSTRACT

This study presents an approach toward the conservation and protection of a historic brass Candelabra housed in the French Institute of Oriental Archaeology in Cairo, which dates from approximately 1815-1830 AD. Recognizing the widespread use of brass alloys comprising copper and zinc in ancient artifacts, due to their aesthetic appeal and corrosion resistance, the research focuses on the importance of advanced preservation techniques to combat environmental degradation in such alloys. The artifact's surface was subjected to detailed analytical investigation utilizing a combination of techniques, including portable light microscopy, scanning electron microscopy (SEM), X-ray diffraction (XRD), and portable X-ray fluorescence (XRF). The analyses revealed significant surface alterations, including copper corrosion products and morphological alterations indicating surface loss and deformation due to exposure to pollutants, humidity, and chloride ions. Furthermore, the thermal effects generated by the repeated use of the candlestick contributed to its surface deterioration, leading to a decline in its physical and mechanical properties and increasing its structural fragility. Conservation treatment of the Candelabra was performed through mechanical cleaning, which included the use of various brushes, scalpels, an ultrasonic technique, and spatulas to effectively remove dust that adhered to corrosion products covered by wax. Chemical cleaning involved immersion of the object in Rochelle salt solutions, then in distilled water to remove the chemicals, followed by ethyl alcohol rinses. Then, the surface was prepared by dry heating in order to apply the protective layer 0.5% BTA + 5mg TiO_2. Benzotriazole forms protective films on brass alloy, while TiO_2 nanoparticles enhance stability, UV resistance, antimicrobial activity, and coating uniformity for improved corrosion protection.

INTRODUCTION

Brass alloy was frequently used in the production of a wide range of historic artifacts. Its ability to replicate decorative features and details of molds while resisting corrosion made it a material of choice for craftsmen, who approved of its resemblance to gold in luster and color, meeting their needs. This is evidenced by the large number of brass alloy artifacts preserved in Egyptian museums and storages, as well as items still being uncovered to this day. This offers an indication of craftsman being fully aware of how to employ tools used in the manufacturing process, as well as the properties of these metals and their alloys, even in the ancient past. Tomb scenes, such as those in the tomb of Vizier Rekhmira in Thebes (18th Dynasty), depict the craftsmen's knowledge of the different methods and stages used in the metal crafting and shaping processes during the pharaonic period, when objects made of copper and their alloys were produced for use in almost all aspects of human life and activity. The first metal to be commonly used by humans was indeed copper and its alloys. The two main categories of alloys are those produced when copper (Cu) combines with tin (Sn) to form a bronze alloy and those containing zinc (Zn), known as Brass (Godfrey & Gilroy, 2017, pp.443–469).

Brass alloy (yellow copper) was used in metalworking in more recent times, as it has a high temperature tolerance. For 'copper + zinc', the melting point is 1000°C when the zinc content reaches 20%, whereas when this percentage reaches 60%, the melting point drops to 833°C (Schorsch, 1988). Most ancient copper–zinc alloys were produced by the cementation process and exhibit a yellow color (Saleh, 2017, p. 551). There are many types of Brass Alloys, with Copper-Zinc (standard Brass) being the most prevalently used alloy as it is suitable for manufacturing and has moderate strength and corrosion resistance (Davis & ASM International, 2001, pp. 225). Leaded Brass (Copper-Zinc with Lead) offers improved machinability, which is suitable for precision components requiring finishes with a smooth surface (Lu et al., 2020, p. 8156).

At the beginning of the nineteenth and twentieth centuries, artists often attempted to achieve color effects on metal surfaces by applying a color layer with patina in order to add aesthetic values as well as being a sensory element through which the artist could express his ideas (Runfola, 2014, p.123). Corrosion of metal alloy objects may occur in museums and storerooms when they are exposed to contaminated air, dampness, chloride, or acid. Acetic acid from wooden crates, textiles, paper, woolens, and wall paints can also corrode metals in exhibition cases and storage rooms (Rocca et al., 2004, p.656; Kontozova-Deutsch et al., 2011, p. 1433). Corrosion is an irreversible interfacial interaction that occurs when metal is exposed to its environment (Costa & Urban, 2005, pp. 50–52). This can disfigure metal alloy artifacts, and the greenish color corrosion products of copper chloride are formed (Oikawa et al., 2005, p. 365). Bronze disease, a form of pitting corrosion, is characterized by the dissolution of the metal substrate beneath the oxide layer (anodic area), while corrosion products accumulate above the Cu_2O film (cathodic area) (Ahmed et al., 2021, p. 151). Understanding the morphology and mineralogy of corrosion products, the cause and mechanism of corrosion, and the types of corrosion products of selected objects can help to identify the ancient manufacturing processes used to produce the selected objects (Ryhl-Svendsen, 2008, p. 287). It can also indicate physical and mechanical processes such as smelting, abrasion, or mechanical fracture, as well as chemical reactions like oxidation and reduction (El-Shamy & Zohdy, 2015, p. 59).

The corrosion behavior of brass exhibits relatively good resistance to atmospheric corrosion and mild environmental conditions; however, it can undergo various forms of degradation. These forms can vary widely based on alloy composition and thermomechanical processing. Alloys can be softened or hardened via cold working or heat treatment to meet specific strength and ductility requirements (Davis & ASM International, 2001, pp. 231–235).

Understanding the causative factors and mechanisms underlying corrosion, along with implementing appropriate preventative strategies, is crucial for safeguarding brass artifacts from degradation and ensuring their preservation for future generations (Fjaestad et al., 1997). Consequently, it is vital to determine the environmental conditions in which these objects are stored or displayed. Maintaining optimal temperature, humidity, and air quality can significantly reduce the risk of corrosion (El-Shamy et al., 2016, p. 1). Moreover, regular cleaning and maintenance are essential to prevent the buildup of dirt and contaminants that may accelerate corrosion processes (Abdel-Karim et al., 2018, pp. 2-10). Careful handling of antique brass objects is also imperative to avoid physical damage that could predispose them to corrosion (Abbas et al., 2021, pp. 337–385).

Application of nanomaterials to improve corrosion inhibition represents a promising advancement in protective coating technologies. Their unique properties, such as increased active surface area with metal substrates, elevated reactivity, and superior mechanical attributes, enable nanomaterials to significantly enhance the efficacy and durability of anti-corrosion systems (Farag, 2020). With enhanced dispersion and homogeneity, when incorporated into coatings, nanomaterials disperse more uniformly, leading to improved barrier properties and minimizing defects that serve as initiation points for corrosion (Wang et al., 2018). Creating robust protective layers, nanomaterials can form thin, flexible layers that prevent the passage of corrosive ions, thus safeguarding the underlying metal (Li et al., 2017). In harsh environments, the integration of nanomaterials increases the resistance of coatings to aggressive environmental factors, such as acids and salts, thus extending their protective lifespan (Rifai et al., 2015). With antioxidant and anti-corrosive properties, certain nanomaterials, like silver nanoparticles and carbon-based nanostructures, possess intrinsic antioxidant activities that mitigate oxidative corrosion processes (Rodrigues et al., 2019).

Chemical characterization of the corrosion products is determined by the type of substrate and the environment in which it is exposed (Walker & Hildred, 2000, pp. 217–219). After obtaining the samples, techniques, including a USB light microscope, XRD, portable XRF, metallographic, and SEM analyses, were used to identify the microstructure and phase analysis of the candelabra. Conservation procedures of the candelabra were performed by mechanical and chemical treatment, followed by the application of a protective layer of 0.5% BTA + 5mg TiO_2.

MATERIALS AND METHODS

The applied research study was conducted on a historical candelabra housed at the French Institute of Oriental Archaeology in El-Monira, Cairo, dating to 1815-1830 AD during the reign of Charles X of France. The candelabra comprises 55 pieces (see Figs. 1–3), where the item was dismantled and its parts detached from each other. It is composed of six lights supported by scroll arms. The item features acanthus terminal accents on reeded standards, mounted on a tripod base with paw feet, and includes an incurvate plinth (LiveAuctioneers, n.d). Its dimensions are approximately 24 inches in height and 10 inches in diameter. The artifact exhibits areas of green oxidation and patina, indicating age and exposure to environmental elements (LiveAuctioneers, n.d).

 DOI: 10.21608/SHEDET.2025.374045.1293

Fig. 1. The candelabra study case housed at IFAO

Fig. 2. Some parts of the candelabra before treatment

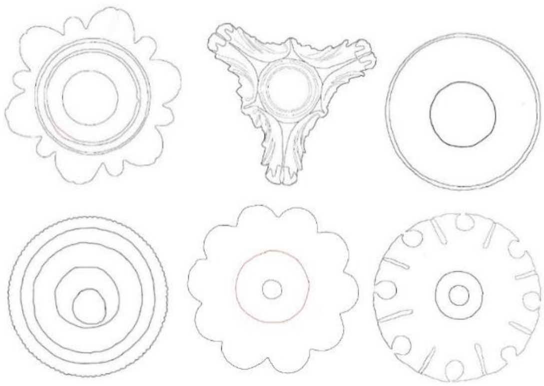

Fig. 3. Drawing of the parts in Fig. 2 using Photoshop

Sampling

Samples were collected from both the upper and lower sections of the candelabra to analyze the morphology and chemical composition of the brass surfaces. The samples were examined using a portable light microscope (LM) and a scanning electron microscope (SEM). Elemental analysis was performed with portable X-ray fluorescence (XRF), while X-ray diffraction (XRD) was employed to identify corrosion products through powder diffraction after sample preparation.

Metallographic analysis involved preparing specimens in three stages: (1) embedding it in epoxy resin followed by polishing with abrasive paper to achieve a smooth surface; (2) further polishing on a bauxite slurry pad and with a soft cloth; and (3) rinsing with warm water, gentle wiping with ethyl alcohol, drying, and etching with ferric chloride to reveal microstructural features. Post-etching, samples were rinsed with ethyl alcohol to remove residual etchant and prepared for microscopic examination. These techniques facilitated a comprehensive evaluation of corrosion products and metallographic characteristics.

Benzotriazole

BTA (Benzotriazole) is one of the most well-known corrosion inhibitors for metals on general surfaces, being used specifically for protecting copper and its alloys from corrosion, particularly on heritage artifacts. Its popularity is attributed to its effectiveness in safeguarding copper from corrosion. It is a cyclic compound containing three nitrogen atoms (El Ibrahimi et al., 2020). For many decades, BTA has been employed as an anti-corrosion agent for copper and its alloys due to the strong corrosion-inhibiting properties of benzotriazole and its ability to form a protective film over copper. This film remains passive unless subjected to fractures. The process involves the formation of cuprous benzotriazole when the metal is immersed in a benzotriazole solution, which subsequently oxidizes to copper benzotriazole (Milošev et al., 2015; Li et al., 2017). Benzotriazole is also utilized at a concentration of 3% in ethanol to protect brass and bronze, where it proves highly effective in treating bronze disease (Ghoniem, 2014).

NANO TIO_2

Nano titanium dioxide (TiO_2) is generally considered one of the materials suitable for the protection and preservation of metal artifacts against corrosion and photoreactive activity (Rahimi & Doostmohammadi, 2019, p. 10). Nano-sized TiO_2 particles are semiconducting metal oxides with unique properties. They are characterized by their low cost, non-toxicity, chemical corrosion resistance, and resistance to ultraviolet (UV) radiation and degradation caused by organic pollutants. Additionally, TiO_2 nanoparticles help prevent biological deterioration (Elsayed, 2025, pp. 378–379). Studies have demonstrated that certain inorganic nanomaterials possess the ability to reduce friction and enhance the resistance of polymers. Moreover, nanomaterials have shown potential to improve the mechanical and surface properties of coatings used for metal protection. Nano coatings containing titanium dioxide exhibit the ability to resist photodegradation under UV radiation, owing to their toxicity and their antimicrobial properties, including inhibition of bacteria and fungi (Liu et al., 2021; Meenatchisundaram et al., 2022).

USB Light Microscope

A (MUSTCAM) USB digital microscope, LCD digital microscope, was used for examination of the surface of the candelabra, as it has a magnifying power of up to 1000x. It is the simplest type of microscope to use.

Metallographic Microscope

The BX Series Upright Metallurgical Microscope at Tabbin Institute for Metallurgical Studies (TIMS) was employed for the investigation of the producer's techniques, and the manufacturing and corrosion phases. It involves preparing a small sample of the material, which is then polished, etched, and studied under a microscope.

X-Ray Diffraction (XRD)

X-ray Diffraction (XRD) was performed using the PAN analytical X-Ray Diffraction equipment model X′Pert PRO with Secondary Monochromator, Cu-radiation (λ=1.542Å) at 45

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K.V., 35 M.A., and scanning speed 0.04o/sec. which is located at the National Center for Mineral Resources. The diffraction peaks between 2θ =2o and 6٠o, corresponding spacing (d, Å) and relative intensities (I/Io) were measured. Diffraction charts and relative intensities were obtained and compared with ICDD files.

Portable X-Ray Fluorescence (XRF)

A portable X-ray Fluorescence (XRF) machine (SciAps, Inc., Woburn, MA, USA) at the French Institute of Oriental Archaeology (IFAO) was used to identify elements of the candelabra.

Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) Bruker AXS-Flash Detector 410-M-Germany, located at Tabbin Institute for Metallurgical Studies (TIMS), was used to identify chemical structures and morphology of the object's surface.

RESULTS AND DISCUSSION

Light Microscope Examination

Light microscopy is essential for examining objects that cannot be seen with the naked eye, as it has a magnifying power of up to 1000x. and reveals corrosion products through color changes on the surface of the brass (see Fig. 4).

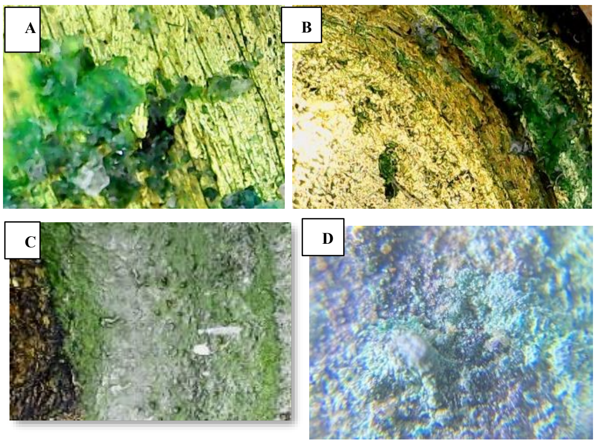

Fig. 4. Forms of corrosion products using a portable light microscope at 800X.

Metallographic Examination (ME)

Metallographic examination is widely used in the manufacturing, engineering and materials science industries to ensure the quality and reliability of metal components and structures (see Fig. 5).

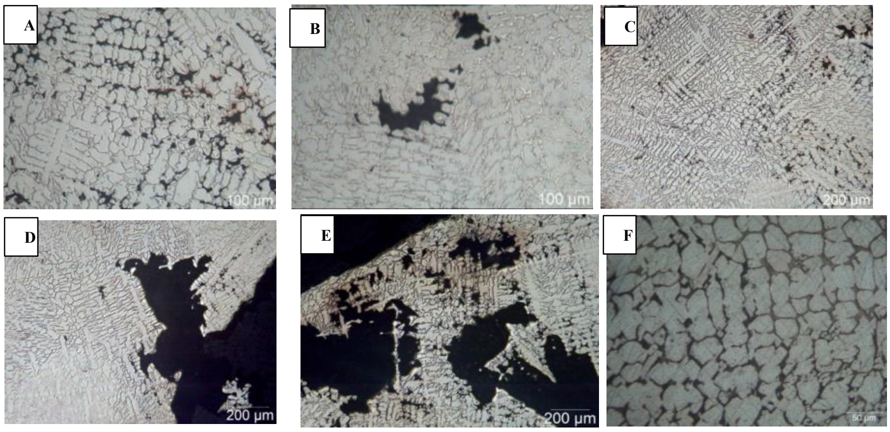

Fig. 5. Metallographic examination for 6 parts of the candelabra:(A) alloy elements overlapping with each other, with loss, 100X; (B) characteristic tree shape of the cast metal, with loss and deformation of the alloy due to corrosion, 100X; (C) some pitting caused by damage 200X; (D) some voids in the alloy caused by a manufacturing defect 200X;(E) alloy deformations under the microscope, and the extent of the loss, at 200X; (F) copper-zinc alloy in the brass alloy and the presence of the dendritic shape, 50X.

Scanning Electron Microscopy (SEM)

SEM examination of brass alloys detected that this alloy has an α-phase and β-phase microstructure, with varying proportions depending on zinc content. They are classified by phase: alpha brass (up to 35% zinc), (α+β) brass (35-46.6% zinc), and beta brass (46.6-50.6% zinc). Alpha brass is ductile, easily cold-worked, and corrosion-resistant, making it useful for artifact preservation. The brass alloy, containing 33.65% zinc, falls within the alpha brass category, being characterized by a homogeneous microstructure without β phase (Megahed et al., 2025). The SEM examination can reveal the surface morphology before conservation procedures. The coating of wax appears to be particularly thick, denoting substantial use of the item (see Fig. 6).

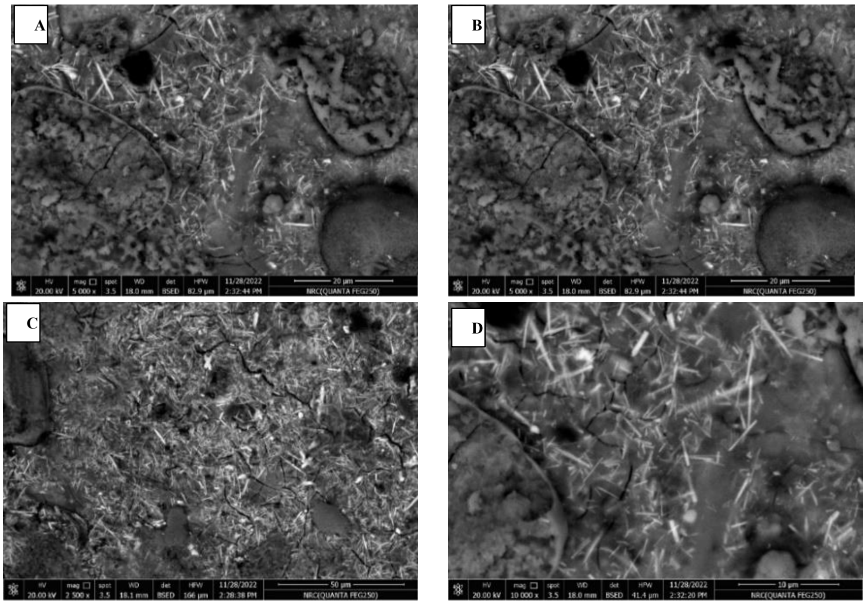

Fig. 6. (A) Wax residue, cracks and fissures in the surface layer of the metal, and some corrosion; (B) waxy residue, cracks, a dense layer of dirt on the surface of the metal, and some corrosion; (C) cracks, surface pitting, and waxy residue; (D) waxy residue mixed with dust and cracks in the surface layer of the metal.

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X-Ray Diffraction Analysis (XRD)

XRD analysis of the corrosion products on the selected objects revealed the presence of major compounds, such as atacamite, paratacamite, cuprite, and malachite, which indicates significant copper corrosion. Minor compounds such as natanite, larnite, and halite were also found, suggesting the effects and presence of environmental factors and additional copper oxide minerals (see Figs. 7–12 & Table 1). Determining the composition of corrosion products is critical for maintenance and restoration efforts, guiding the selection of appropriate treatment methods and materials. In addition, these results contribute to a better understanding of corrosion processes in historical and archaeological objects, aiding the preservation of cultural heritage.

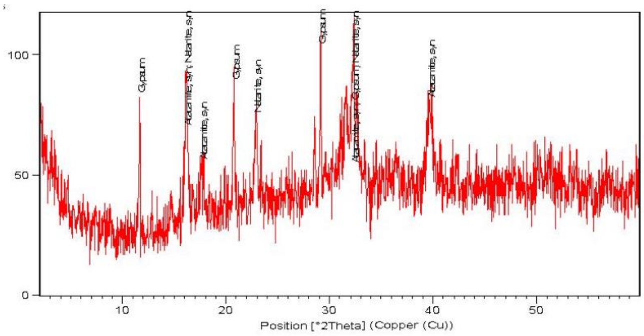

Fig. 7. XRD patterns of sample A

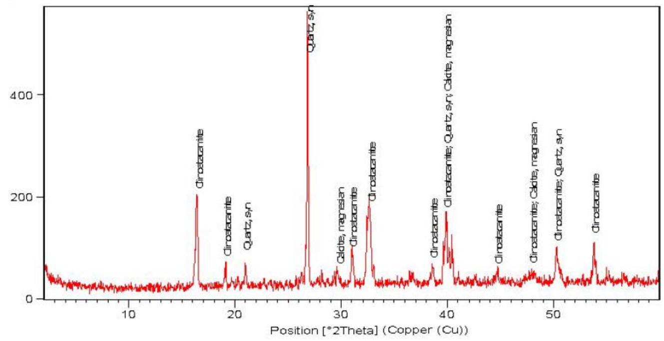

Fig. 8. XRD patterns of sample B

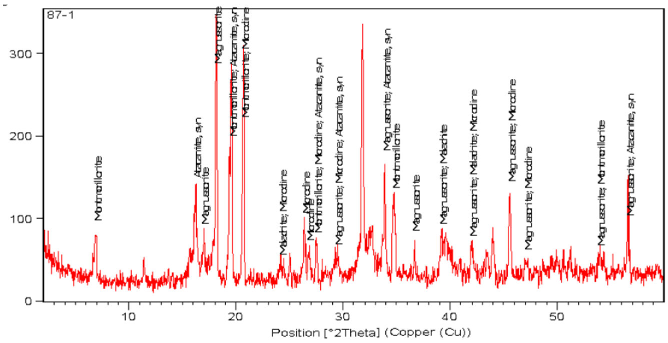

Fig. 9. XRD patterns of sample C

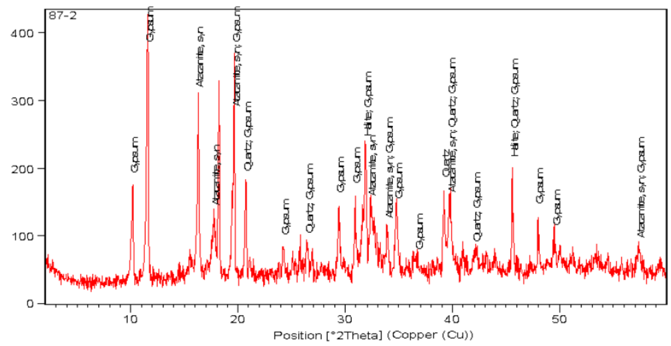

Fig. 10. XRD patterns of sample D

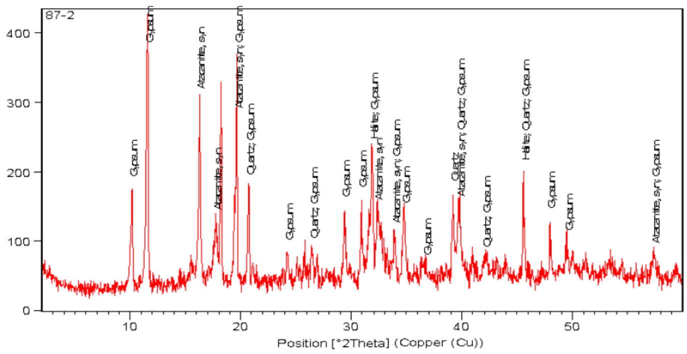

Fig. 11. XRD patterns of sample E

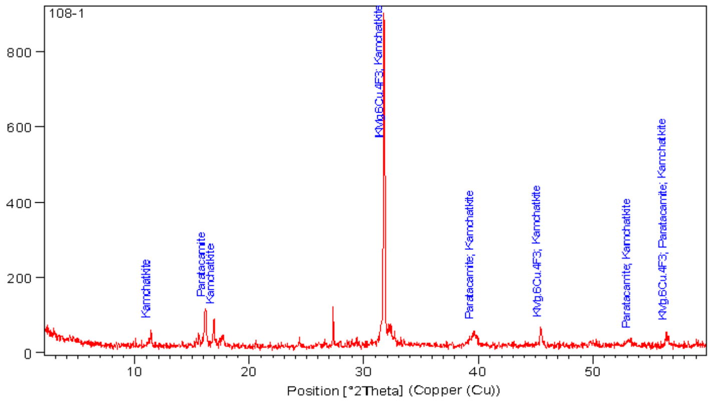

Fig. 12. XRD patterns of sample F

Table 1. Shows the XRD analysis results of the corrosion products of the selected objects.

Samples	**Compounds**		
	Major	**Minor**	**Traces**
Sample A	Atacamite $Cu_2(OH)_3Cl$	Larnite $Ca_2(SiO_4)$	Quartz SiO_2
Sample B	Atacamite $Cu_2(OH)_3Cl$	Natanite Fe (n $(OH)_6$)	-------
Sample C	Atacamite $Cu_2(OH)_3Cl$	Natanite Fe (n $(OH)_6$)	-------
Sample D	Atacamite $Cu_2(OH)_3Cl$	Malachite $CuCO_3.Cu(OH)_2$	Natanite Fe (n $(OH)_6$)
Sample E	Atacamite $Cu_2(OH)_3Cl$	-------	Halite NaCl
Sample F	Paratacamite $Cu_2Cl(OH)_3$	Cuprite Cu_2O	-------

Portable X-Ray Fluorescence Analysis (XRF)

X-ray fluorescence analysis (XRF) is a non-destructive method that was used to determine the elemental composition of six samples from the candelabra. It revealed the presence of both copper and zinc, the two main components of brass alloy (Table 2 & Fig. 13:18). These results not only provide insight into the composition of objects but also contribute to an understanding of their historical context, previous conservation efforts, and the intended preservation of cultural artefacts.

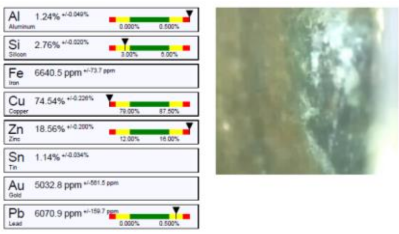

Fig. 13. Sample A

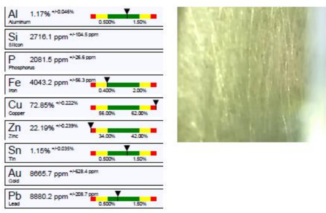

Fig. 14. Sample B

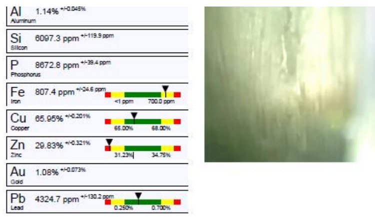

Fig. 15. Sample C

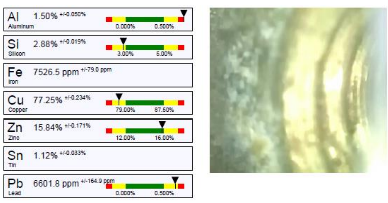

Fig. 16. Sample D

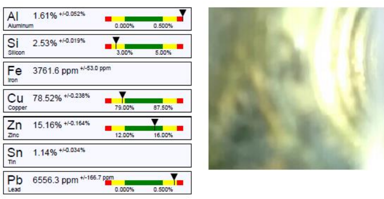

Fig. 17. Sample E

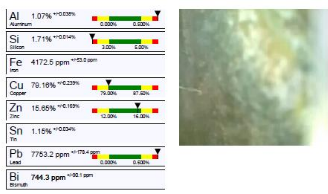

Fig. 18. Sample F

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Table 2. Shows the XRF analysis results of the objects.

Elements / Samples	Cu, %	Zn, %	Pb, %	Fe, %	Sn, %	Au %	Si %	Al %
Sample A	74.54	18.56	0.66	0.66	0.68	0.05	2.76	1.24
Sample B	72.85	22.19	0.88	0 .40	1.15	0.86	0.27	1.17
Sample C	65.95	29.83	0.43	0.87	0	1.08	0.60	1.14
Sample D	77.25	15.84	0.66	0.75	1.12	0	2.88	1.50
Sample E	78.52	15.16	0.65	0.37	1.14	0	2.53	1.61
Sample F	79.16	15.65	0.77	0.41	1.15	0	1.71	1.07

TREATMENT AND CONSERVATION

Following a comprehensive examination and analysis of the artifact, including an assessment of its overall condition, the chemical composition of the brass, and the damage imposed by corrosion, wax residues, and dirt, the artifact underwent a careful cleaning process. Each disassembled component of the candelabra was treated individually to ensure thorough preservation.

Conservation treatment was performed via mechanical cleaning, which included the use of various brushes, scalpels, ultrasonic technique, and spatulas to effectively remove dust adhered to corrosion products covered by wax. Chemical cleaning involved immersion of the object in Rochelle salt solutions, then immersion in distilled water to remove the chemical, followed by ethyl alcohol rinses. Subsequently, the surface was prepared by dry heating for the application of the protective layer 0.5% BTA + 5mg TiO_2, as illustrated in (Fig. 19:22).

Fig. 19. Mechanical cleaning (A and B) using different brushes to remove dust and corrosion; (C) using a scalpel to remove wax and corrosion; (D) Ultrasonic to remove hard corrosion; (E and F) using a spatula to remove old wax from the base.

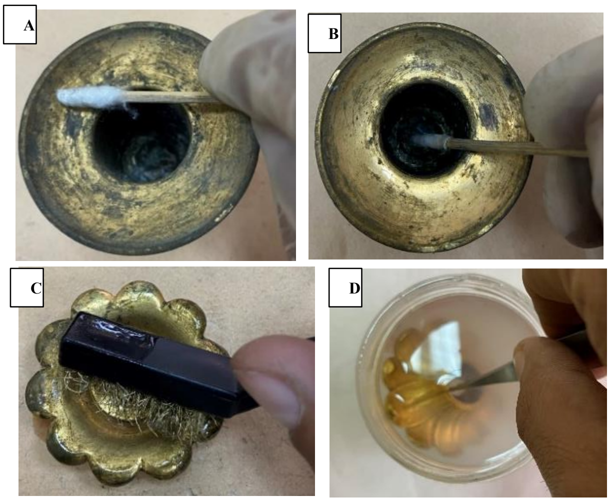

Fig. 20. Chemical cleaning (A and B) using swap after placing it in a Rochelle salt solution to remove corrosion products; (C) using a metal brush to remove corrosion products; (D) putting the object after cleaning into 0.5% BTA + (5mg) TiO_2 for protection.

Fig. 21. Candelabra parts after treatment and protection with 0.5% BTA + (5mg) TiO_2.

Fig. 22. The candelabra after the intervention and protection treatments.

DOI: 10.21608/SHEDET.2025.374045.1293

CONCLUSIONS

This study highlights the effectiveness of incorporating nanomaterials, notably titanium dioxide (TiO_2) nanoparticles, with benzotriazole (BTA) to enhance the corrosion resistance of historic candelabra artifacts. Investigation techniques, including SEM, XRD, and XRF, revealed substantial surface deterioration attributed to environmental factors such as pollution, humidity, and chloride exposure, resulting in the formation of corrosion products, which primarily consisted of copper products. The application of a protective coating formulated with BTA augmented by TiO_2 nanoparticles significantly improved corrosion inhibition, offering evidence for its potential for preserving metallic heritage objects. The nano-enhanced formulation contributed to the formation of a more uniform, adherent, and durable protective film on the artifact's surface. The intrinsic properties of TiO_2 nanoparticles—such as chemical stability, UV resistance, and antimicrobial activity—further enhance the coating's protective capacity, potentially reducing biological deterioration and the effects of photodegradation.

Overall, this study provides valuable insights into innovative conservation technologies that strike a balance between efficacy, safety, and sustainability in the preservation of historical brass objects.

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علاج وحماية شمعدان تاريخي من سبيكة البراس باستخدام بنزوتريازول مُحسَّن بحبيبات نانو أكسيد التيتانيوم محفوظ بالمعهد الفرنسي للآثار الشرقية

محمد صديق

قسم الترميم، كلية الآثار، جامعة الفيوم، الفيوم، مصر؛ قسم الترميم، كلية الآثار والإرشاد السياحي، جامعة مصر للعلوم والتكنولوجيا.

mohamed.sedek@must.edu.eg

رجب أبو الحسن

قسم الترميم، كلية الآثار والإرشاد السياحي، جامعة مصر للعلوم والتكنولوجيا.

raga.sayed@must.edu.eg

صالح محمد صالح

قسم الترميم، كلية الآثار، جامعة الفيوم، الفيوم، مصر.

sms02@fayoum.edu.eg

محمد مجاهد

قسم الترميم، كلية الآثار، جامعة الفيوم، الفيوم، مصر.

mmm03@fayoum.edu.eg

بيانات المقال

تاريخ المقال

تم الاستلام في ٩ أبريل ٢٠٢٥

تم استلام النسخة المنقحة في ١٤ مايو ٢٠٢٥

تم قبول البحث في ١٧ مايو ٢٠٢٥

متاح على الإنترنت في ١٧ يوليو ٢٠٢٥

الكلمات الدالة

سبيكة البراس، النحاس والزنك، الشمعدان التاريخي، بنزوتريازول (BTA)، الجسيمات النانوية (TiO_2)، مانع التآكل، الصيانة

الملخص

تُقدم هذه الدراسة منهجًا شاملاً للحفاظ على شمعدان تاريخي من سبيكة البراس محفوظ في المعهد الفرنسي للآثار الشرقية بالقاهرة، والذي يُقدر تاريخه بين عامي ١٨١٥ و١٨٣٠م تقريبًا. ونظرًا للانتشار الواسع لاستخدام سبيكة البراس، المكونة من النحاس والزنك، في القطع الأثرية القديمة نظرًا لجاذبيتها الجمالية ومقاومتها للتآكل، يُؤكد البحث على أهمية تقنيات الحفظ المتقدمة لمواجهة عمليات التدهور البيئي التي تتعرض لها هذه المواد.

حيث خضع الشمعدان لدراسات تحليلية تفصيلية باستخدام مجموعة من التقنيات المتنوعة، بما في ذلك المجهر الضوئي المحمول، والمجهر الإلكتروني الماسح(SEM) ، وحيود الأشعة السينية (XRD)، وتقنية التحليل باستخدام تفلور الأشعة السينية المحمولة (XRF). وقد كشفت التحاليل عن تغيرات كبيرة على مستوى السطح، تتضمن نواتج من مركبات النحاس، إلى جانب تغيرات مورفولوجية تشير إلى فقدان في السطح وتشوهه الناتج عن التعرض للملوثات والرطوبة وأيونات الكلوريد. علاوة على ذلك، ساهمت التأثيرات الحرارية المتكررة الناتجة عن استخدام الشمعدان في تدهوره، مما أدى إلى انخفاض خصائصه الفيزيائية والميكانيكية وزيادة هشاشتها البنائية.

لترميم الشمعدان وحمايته، أُجريت عمليات تنظيف ميكانيكي باستخدام الفرش ومشارط متنوعة، بما في ذلك استخدام جهاز الموجات فوق الصوتية لإزالة الغبار ومواد التآكل المغطاة بالشمع بشكل فعّال. وتبع ذلك عملية تنظيف كيميائية عبر غمر القطعة في محلول ملح روشيل، يليه غمر في الماء المقطّر لإزالة باقي نواتج المواد الكيميائية، وشطفها بالكحول الإيثيلي. كما تم إعداد السطح من خلال التسخين الجاف لتطبيق طبقة واقية تتكون من ٠,٥% BTA (بنزوتريازول) و٥ ملج جزيئات ثاني أكسيد التيتانيوم النانوي TiO_2. يُعد البنزوتريازول طبقة فاعلة على سبيكة البراس، حيث تساهم خصائصه في تشكيل أغشية واقية، في حين تعزز جزيئات TiO_2 النانوية ثبات المادة، ومقاومتها للأشعة فوق البنفسجية، ونشاطها المضاد للميكروبات، وتوحيد الطلاء لتحسين الحماية من عمليات التآكل.