Art restoration and archaeological material study are inseparately related to scientific investigation and scientific data processing of the information. This reality makes the mentioned field of the most attractive one and a very generous one for professional development.

The e-proceeding, published by INTEGRA NATURA ET OMNIA - INOE, brings together a large part of the contribution to The Third Balkan Symposium on Archaeometry organized in Bucharest on 29 and 30 October 2012, and is following the close related volume that was published by The Kultur Intitute University from Istanbul. The biennial event gathers scientists, conservators, restorers, architects, companies, decision-makers, professors and students involved in projects on all aspects of archaeometry, the application of modern experimental methods and techniques used in investigation, identification and dating of ancient artifacts, as well as related fields of archaeology and art history. Appreciated researchers from multidisciplinary groups, not only from Balkans, have been invited to contribute with keynote speeches and to support the dissemination of recent results. The event continues the tradition of previous symposiums, the first being held in Ohrid - Republic of Macedonia in 2008 and the second in Istanbul – Turkey in 2010.

Special support for present e-proceeding publishing have been received from Dr. eng. Roxana Savastru – general manager of INOE, who was sustaining all initiatives of the Center for Restoration by Optoelectronical Techniques and who permanently, and who generously offers her experience and professional skills.

The editors wish to remind to all participants to The Third Balkan Symposium on Archaeometry that the devoted specialist and initiator of the Balkan Network on Archaeometry – permanently close to each edition organization- is Prof. Prof. Biljana Minceva-Sukarova from Institute of Chemistry, Faculty of Natural Sciences and Mathematics “SS. Cyril & Methoidius” University, Republic of Macedonia.

Proceedings of the

3rd Balkan Symposium on Archaeometry

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Bucharest, Romania

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Chemical and mineralogical study of a Yamur from the Thirteen Century

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Abstract: The aims of this research were the chemical and mineralogical study of a Yamur from the thirteen Century and the study of their conservation degree in order to facilitate the restoration. The Yamur was located in the upper part of the dome of the Conceptionists Convent in Los Pedroches (Cordoba, Spain). The restoration project was carrying out in the Andalusian Institute of Cultural Heritage (IAPH), according to the silver restoration protocol under the commission of the Andalusian Culture Ministry. The Characterization was carry out by Optical Microscopy (OM), X-ray Diffraction (XRD), Inductively Coupled Plasma (ICP) and Scanning Electron Microscopy coupled to energy dispersive X-ray spectroscopy (SEM-EDX). The spheres are made of brass (copper and zinc alloy) with minor proportion of lead. This composition decreases the melting point and allowes the work of the piece. The sphere brass corrosion is due to the presence of cuprite and paramelaconite while the illite found in the deposits could have a soil origin.

1 INTRODUCTION

A yamur is a metal piece that is located on the minarets of Islamic mosques. It consists of three or more spheres, assembled from the highest on the bottom to the lowest on the upper part.

These spheres symbolize the three worlds where God is made known according to the Islamic culture, (mulk, material world, malakut, imaginary world, and yabarut, world of power), representing the perfection of God and the universe (related to circular or spherical shape), and identify the place of prayer in Islamic Culture.

The aim of this work is to study the chemical and mineralogical composition of the Yamur (Figure 1) that is over the Dome of the Convent of Conception, Los Pedroches (Córdoba, Spain).

The three spheres were made of brass, their sizes from the highest to the lowest are: 1.32 m., 0.90 m. and 0.65 m, they are assembled by an iron stem with a flag and a cross on the upper part.

2 MATERIALS AND METHODOLOGY

The first study was the description of the corrosion and the conservation degree of the pieces (spheres, stem, flag and cross).

For this research, 20 samples have been taken from different alloys and corrosion products following the recommendations of the technical commission CNR-ICR NORMAL 3/80 [8].

Metallographic sections have been prepared with $FeCl_3$ in ethanol according (Scott, 1991).

The mineralogical characterization was carried out by an X-ray difractometer brand Bruker (model D8 Advance), using CuKα and an Optical Microscope Leica DM4000M.

The elemental chemical analysis over the stratigraphies of the crusts and deposits have been studied by means of electron scanning microscope JEOL JSM-5400, with x-ray energy detector, Inca X-sight. The chemical composition has also been analyzed by inductively coupled plasma (ICP) with a Horiba Jobin Yvon 2.

Figure 1: *Yamur before restoration. Image: Eugenio Fernández Ruiz (IAPH)*

3 RESULTS AND DISCUSSION

3.1 *Damages of the Spheres*

The spheres presented a high level of weathering with deposits, even bird droppings, iron stains, deformation, losses, fissures and oxidation products of copper among other pathologies (*Fig. 2-3*).

Figure 2: *Bird droppings, deposits and green corrosion product on a Sphere of the Yamur before restoration. Image: Eugenio Fernández Ruiz (IAPH)*

Figure 3: *Holes, fissures and green corrosion product on the Spheres of the Yamur before restoration. Image: Eugenio Fernández Ruiz (IAPH)*

3.2 *Damages of the Iron pieces*

The iron pieces that support the sphere contain in the upper part a flag and a cross.

These iron pieces were very rusty and deformed. The flag cross and structural support presented a high level of corrosion and deposits of different origin (*Fig. 4*).

Figure 4: *Deformation and iron corrosion products on the iron structure of the Yamur before restoration. Image: Eugenio Fernández Ruiz (IAPH)*

3.3 *Chemical and Mineralogical Study of the Spheres*

The spheres are made of brass, copper and zinc alloy (65:30), with a 2% w/w of lead according to ICP analysis. The addition of lead in the alloy decreases the melting point and facilitates its work.

The cross section of the brass structure treated with $FeCl_3$ in ethanol is alpha-phase with equiaxed grains with bands caused by cold working (*Fig. 5*).

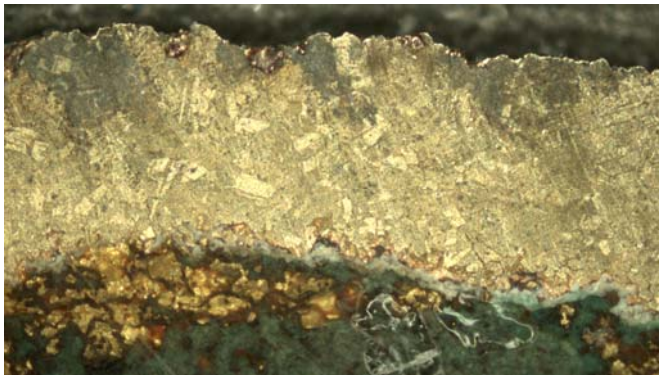

Figure 5: *Optical microscopy photograph of the cross section of a brass sample treated with $FeCl_3$ in ethanol (X5).*

The XRD study of the patinas discovered the presence of cuprite (Cu_2O), paramelaconite (Cu_4O_3), and Illite ($K(AlFe)_2AlSi_3O•10(OH)2H_2O$).

Cuprite and paramelaconite (*Fig. 6*) are generated by sphere brass surface corrosion due to the atmospheric conditions while the Illite found in the deposits could have a soil origin.

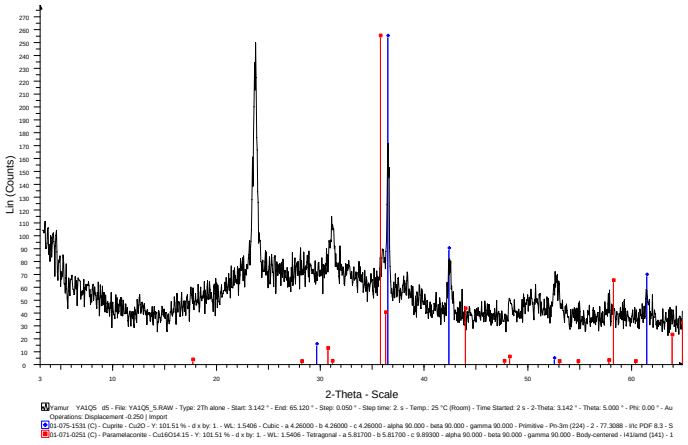

Figure 6: *Cuprite (Cu_2O) and paramelaconite (Cu_4O_3) detected by XRD on the spheres.*

These brass corrosion products are mixed with calcite ($CaCO_3$), anortite ($CaAl_2Si_2O_8$), quartz (SiO_2) and gypsum ($CaSO_4·2H_2O$) that could have an atmospheric origin and whewellite ($Ca(C_2O_4)·(H_2O)$) likelihood due to biological microorganism.

3.4 *Chemical and mineralogical study of the iron pieces*

The flag, cross and stem are covered with a patina mainly of iron oxyhydroxides goethite (FeO(OH), *Figure 7*) and lepidocrite (FeO(OH)/ $Fe_2O_3.H_2O$) with punctual corrosion of lead: cerussite ($Pb(CO_3)$) and hydrocerussite ($Pb_3(CO_3)_2(OH)_2$)).

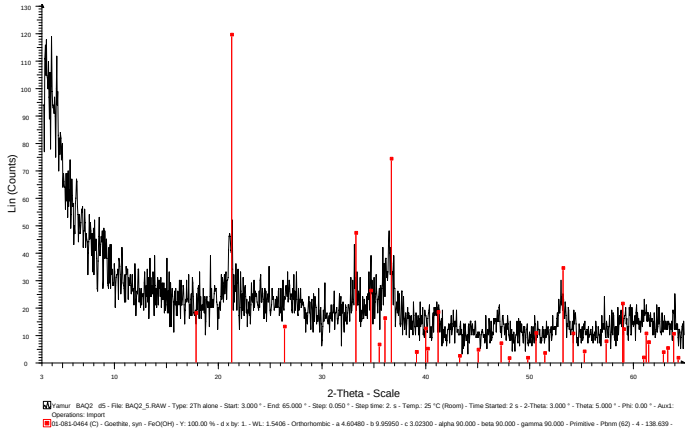

Figure 7: *Goethite (FeO(OH)) detected by XRD on the iron structure.*

The stem has the worst conservation degree with a lot of corrosion products and deformation that could be induced by a galvanic process by the contact between the iron and brass.

3.5 *The restoration*

The restoration consists on the following steps:

- Removing the components (spheres and iron pieces)
- Mechanical and hand-made cleaning of the elements of iron and brass.
- Chemical cleaning and neutralizing of chemical products
- Stabilization of metals by corrosion inhibitors (*Figure 8*).
- Volumetric reintegration with reversible neutral supports to stabilize the pieces.

Figure 8: *Stabilization of metals by corrosion inhibitors on a Sphere of the Yamur during restoration. Image: Eugenio Fernández Ruiz (IAPH)*

The restoration results are shown in *Figure 9*. The stability of the restored structure allows having again their function on the Dome of the Convent of Conception at Los Pedroches (Córdoba, Spain).

Figure 9: *Yamur before and after restoration. Image: Eugenio Fernández Ruiz (IAPH)*

4 CONCLUSION

The Yamur is made of brass, copper and zinc alloy (65:30), with a 2% w/w of lead according to ICP analysis. The addition of lead in the alloy decreases the melting point and facilitates its worked. The brass structure is alpha-phase with equiaxed grains with bands caused by cold working.

The corrosion products depend on the composition of the alloys and the environmental conditions.

Sphere brass surface corrosion generates cuprite and paramelaconite due to the atmospheric conditions while the Illite found in the deposits could have a soil origin.

The flag, cross and stem are covered with a patina mainly of iron oxyhydroxides goethite and lepidocrite with punctual corrosion of lead: cerussite and hydrocerussite. These brass corrosion products are mixed with calcite, anortite, quartz and gypsum that could have an atmospheric origin and whewellite likelihood due to biological microorganism.

The stem has the worst conservation degree with a lot of corrosion products and deformation that could be induced by a galvanic process between the iron and the brass.

This research evidences the need to use corrosion inhibitors in the restoration to slow down the corrosion when the Yamur return to their location in the Dome.

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