

## **Analysis, Technical Investigation and Conservation of a Painted Enamel Qajar Pendant**

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**Summary:** This research aims at investigating the chemical composition and the technical features of a painted enamel Qajar pendant. This investigation was followed by cleaning and conservation of the pendant. Optical light microscope (OLM), scanning electron microscope (SEM) combined with energy dispersive X-ray spectroscopy (EDX), Fourier transform infrared spectroscopy (FTIR) and X-ray fluorescence analysis (XRF) were used respectively for imaging and micro-analysis of the object. The study proved that the pendant is made of gold foils, round and half-round gold wires of different sizes and carats. Different forming and decorative techniques were found to be used for the manufacturing of the pendant: repoussé, granulation, filigree, chasing, stamping, painted and cloisonné enameling. The study also proved that the central quatrefoil ornament is not solid metal, containing a wax core.

### **Introduction:**

Qajar art is the art of Persia during the rule of the Qajar dynasty, from 1794 to 1925. Enamel working on metals was one of the significant forms of art in Isfahan, Iran. Painting with enamels, or what is referred to as painted enamel was first developed in Limoges, France in the 15<sup>th</sup> century and later transferred into Iran and other parts of the world. The examples of enamels that survived reflected the interest of Iranian artists in this art since the Achaemenian dynasty (c. 550–330 B.C.). Qajar enamels are characterized by figurative scenes including portraits of youth and

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lovers. These were similar in style to the oil paintings of the same period. Luxury, prevalent costume and jewelry styles are apparently documented in such portraits and scenes [1-7]. There are quite few examples of Qajar painted enamel pendants which survived and are now exhibited in museums world wide.

#### **The case study:**

The case study presented in this research is a Persian enameled gold pendant. It belongs to the museum of the Faculty of Applied Arts at Helwan University, Egypt. It was given the identification number 49/1 in the museum records although the details about its source and date are not specified. Figures 1a and 1b show the obverse and reverse of the pendant. Lack of information affected the documentation and authentication processes. For its authentication, a survey was needed to identify possible parallels showing a similar style, technical features and material structure. Microchemical analysis was also required for proper identification and documentation of the object. Examination and investigation were required as well to identify the state of preservation before undertaking any further conservation intervention. From the conservation point of view, the principle of minimal intervention is uppermost not only because of the condition of the object but also for ethical guidelines.

#### **Experimental:**

The surface of the object was examined by optical microscopy to show details and deterioration aspects. A digital caliper was used for measuring the length, the width and the diameter of the different parts of the pendant. A tiny sample of filling material (the core) was examined by optical light microscope (OLM) before analysis. The sample was also analyzed by Fourier transform infrared spectroscopy (FTIR) using a Jasco-460. The sample was first ground and pressed in KBr pellets. The analysis has a spectral range from 400 to 4000  $\text{cm}^{-1}$  with a maximum resolution of 4  $\text{cm}^{-1}$ . All spectra were recorded in transmission mode.

Scanning Electron Microscopy (SEM) imaging and energy-dispersive X-ray spectroscopy (EDX) micro-chemical analysis of the counter enamel were undertaken using a Philips XL 30 combining SEM with EDX. The sample taken for this analysis was first coated with carbon. The main detection limits of EDX measurement are 0.2% for Cu, 0.5% for Au and 0.1% for Ag. Data at or below these limits are not significant.

A portable Niton XLt 700, version 4, X-ray fluorescence (XRF) analyser was used to determine the composition of different parts of the metal and enamel composition of different colors of the portrait. The Certified Reference Material used for the measurement was 35 EN-04292005-IARM-P. The error was not more than  $\pm 0.2\%$  for Cu,  $\pm 0.4\%$  for Pb,  $\pm 0.1\%$  for Sn,  $\pm 0.4\%$  for Ag,  $\pm 0.09\%$  for Au,  $\pm 0.1\%$  for Sb,  $\pm 1.6\%$  for Fe and  $\pm 0.05\%$  for Zn. The obtained results are the average of two measurements.

Precipitated calcium carbonate in denatured ethyl alcohol, 100% pure cotton cloth and ethyl alcohol were used for cleaning, polishing and wiping off the metal surface, respectively.

#### **Results and discussion:**

A parallel was found by the author in an enameled gold pendant from the Metropolitan Museum collection (figure 2). It is dated to the 19<sup>th</sup> century and belonged originally to Iran. It was fabricated from metal sheet and half-round wire and enameled on both obverse and reverse sides. The common shapes of such enameled plaques are round and elliptical and they were usually set into non-ornamental objects such as water pipes. However, this pendant has engravings on its back and it's thought to have been used as talisman [6].

A comparison between the proposed parallel pendant and the present case study reveals that the object under investigation is similar in style and can be identified as a Qajar pendant which is full of decorations (figures 1a and 1b). It weighs 25 g and its dimensions are 11.5 cm long, 4.5 cm maximum width and 5 mm

thick. The pendant consists of three elements: the chain, the inverted crescent element and the central quatrefoil ornament with seven dangling discs attached. In order to investigate the quality of the metal wires and foil, a non-destructive XRF microchemical analysis was undertaken for the metal parts and the enamel. The results are given in table 1.

**Table 1: Analysis results of the metal parts and enamel using XRF spectroscopy.**

Analyzed part	Chemical composition wt%														
	Au	Ag	Cu	Sn	Pb	Sb	Fe	Cd	Zn	Bi	In	Se	Ni	Co	Mn
Metal reverse /repoussé work	60.23	32.93	4.41	1.49	0.04	0.29	0.06	0.06	0.07	0.16	0.15	0.01	0.01	0.00	0.02
Metal /net-work	67.88	21.07	8.78	1.00	0.00	0.10	0.20	0.05	0.14	0.16	0.11	0.35	0.01	0.01	0.09
Metal/ chain	65.30	25.50	6.92	0.91	0.01	0.09	0.11	0.07	0.04	0.11	0.03	0.2	0.02	0.01	0.11
Enamel rose color	15.51	7.87	2.17	7.76	63.61	0.39	1.69	0.06	0.07	0.25	0.00	0.05	0.00	0.38	0.00
Enamel black color	22.11	6.85	2.73	6.35	50.80	0.16	6.80	0.00	0.31	0.47	0.00	0.21	0.55	0.46	2.14

From the obtained results it is evident that the pendant was made of a gold alloy (gold, silver and copper ternary alloy). Different quantities of constituent metals were used to produce foils, round and half-round wires of different gauges. Different forming and decorative techniques were used for the fabrication of the pendant. Repoussé was used for decorating the reverse side of the central ornament by embossed flowers (figure 1b) and chasing was used to decorate the sides and contour strip of the painted enamel portrait. Net work was used to make an up side down dangling crescent. This was decorated by granulation. Stamping was used to decorate six small discs by an eight leaves flower pattern punch. These small dangling circular discs were soldered to small loops after stamping. Painted enamel technique was used for the portrait of the beautiful young woman standing in front of a colored geometric background, most probably resembling a window and wearing a jeweled red

dress. Cloisonné enamel was also used to inlay a small disc dangling from the central lower part of the pendant with white, pink, green and blue colors.

Segments of the manufacture such as foil and wire production could have been independent operations. High carat gold was cut out, embossed, painted with enamel and fired before it was assembled into its place.

It is known that enamels are composed principally of silicate formers, fluxes such as borax, stabilizers, and different oxides as colorants, giving the enamel its color [8]. The enamel is mixed with oil and then painted on the object to give an effect very much like that of oil paints. To apply painted enamel on a gold foil it is usually turned to slightly convex, then a layer of flux called counter enamel is applied on the metal back to reduce stresses arising from different expansion coefficient of metal and enamel. The metal surface is then covered with a uniform layer of enamel that, when fired, produces a background for the drawing. The painting is applied color by color using a paint brush. The highest flowing temperature enamel is fired first and followed by lower flowing temperature enamels. This can prevent colors diffusion at later stages. Multiple firing is required to fixate the colors, sometimes up to 20 rounds of firing are need for a complete project. The firing temperature can be up to 800 °C (1500 °F) [9].

Microchemical analysis using XRF (table 1) revealed that the finest gold alloy was used for making the filigree crescent ornament, SEM examination and EDX analysis results (figures 3 and 4) revealed that enamel was applied on gold of about 20 carats. High carat gold is normally required for more exquisite enameling [10]. To this fine gold foil, opaque white counter enamel was applied on the back (figure 3). EDX analysis results of the counter enamel (figure 4) in combination with FTIR analysis results (figure 5b) revealed that both calcium and carbonate group are present. This may indicate the use of either lime containing counter enamel, such as soda-lime,

or calcium antimonite opaque white enamel. Wax was identified by FTIR analysis of the pendant core (figures 5a and 5b). This core may have not only supported the gold foil during assembly but also gave the figurative motif further structural support. The added weight by the core may have played an aesthetic role by allowing the pendant to dangle or to sway, and an economic role by reducing the quantity of precious materials used for the manufacturing of the pendant.

The analysis results of the black color enamel reveal the presence of iron, while those of the rose color enamel reveal the presence of iron and copper. The oxides of these elements are responsible for the produced colors [11].

It was difficult for the author to get meaningful analyses results of other enamel colors in smaller areas using the available portable XRF apparatus.

The chemical composition of enamels can indicate the period of production and authenticate the object [8, 11-14]. The chemical composition of medieval enamels is similar to that of the Roman period, consisting of soda-lime glass mixed with opacifiers such as tin or antimony oxides. In the 18<sup>th</sup> and 19<sup>th</sup> centuries, the chemical composition was basically lead-potash glass with arsenic oxides opacifiers. XRF could not detect some elements such as sodium and magnesium so it prevented identification of flux but the presence of tin (Sn) as an opacifier is in agreement with literature on medieval production [12, 13]. Moreover, the analysis results of enamel using XRF revealed that lead (Pb) content is very high (table 1), this result is in agreement with the assumption that lead-potash glass was used for the painted enamel, as lead became an almost universal additive to enamels by the 19<sup>th</sup> century [14]. Accordingly, the pendant is suggested to date back to the 19<sup>th</sup> century, but this is in contradiction with the analysis result of the counter enamel so dating of the pendant still needs further work for archaeologists to explain these results.

### **Cleaning and conservation:**

Both of the vitreous material and the metal support were taken into account when carrying out the conservation treatment. The enamel was in a good condition, presenting no cracks, although there were some missing parts of the green circle that outlines the portrait. At first, the whole pendant was cleaned from dust with a soft brush. The metal parts of the object were cleaned using precipitated calcium carbonate in denatured ethyl alcohol. This was applied using 100% pure cotton cloth for cleaning and polishing metal surface (figures 6 and 7). Calcium carbonate proved to be efficient, abrasive and least likely to scratch soft metal surfaces [15]. The metal surface was cleaned after polishing using ethyl alcohol and cotton swaps. The object was allowed to dry air immediately after cleaning, meanwhile avoiding leaking of the carrier fluid to the inner core. Dirt and grime in cloisonné enamel was mechanically removed using a soft, pointed wooden stick, followed by cleaning by ethanol which also served to degrease the metal. 3% of Paraloid B72 in acetone was applied on the whole surface to give a transparent protective thin film. Paraloid B72 is used in preference to other protective coating materials as it ensures protection and provides consolidation and visual improvement of the surface [16]. Moreover, it is non-staining for enamel and less likely to stain metals. Following the work of Ryan *et al.*, an airtight Plexiglass showcase conditioned to 40% RH, using silica gel (PRO Sorb) was designed for temporary display of the pendant [17]. This Plexiglass microenvironment showcase was intended to provide a stable RH environment (figure 8) which is important for the preservation of metal enamel composites [18].

### **Conclusion:**

Optical examination enabled full technical description and detailed documentation of a painted enamel gold Qajar pendant of The Faculty of Applied Arts at Helwan University, Egypt. The pendant could have been used as head of a water pipe. Micro-chemical

analysis enabled to identify the metal as being a gold alloy and two enamel colors compositions. The study also proved the presence of a wax core. The object was cleaned and coated with a transparent Paraloid B72 protective film and isolated in a controlled relative humidity microenvironment showcase. The showcase was made of Plexiglass to fit the size of the object. These temporarily procedures were meant to minimize the effect of high and fluctuating relative humidity until the museum renovation works take place.

### **Biography:**

Wafaa Anwar Mohamed, PhD, associate professor of metals conservation at the Conservation Department, faculty of Archaeology, Cairo University. Her principal interests focus on the conservation of metal objects, Examination and conservation of metals-organic and inorganic -composite artifacts and metal technologies in ancient Egypt. She has been teaching conservation of metals for undergraduate and graduate conservation students and supervises master and doctoral conservation researches since 2000. In addition to teaching she has recently took up her position as head of the Quality Assurance Unit at the Faculty of Archaeology, Cairo University, Egypt.

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Fig. 1a: Obverse side of the investigated pendant.



Fig. 1b: Reverse side of the investigated pendant.



Fig. 2: The Metropolitan museum parallel pendant. (<http://www.metmuseum.org/toah/works-of-art/20.106.2>).

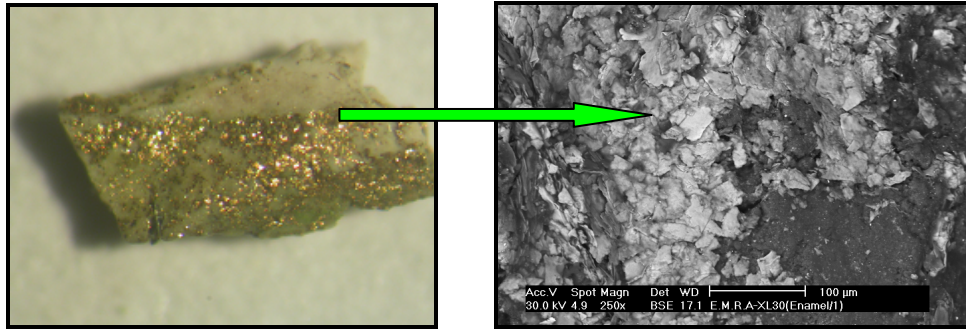


Fig. 3 High-carat gold foil with counter enamel in the back (left) as imaged by SEM (right).



Fig. 4 SEM image and EDX analysis result of gold foil.



Fig. 5 a Core material inside the quatrefoil central ornament

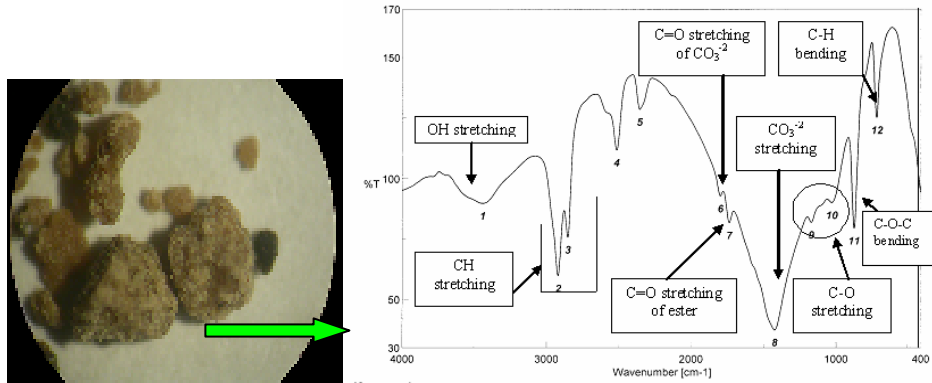


Fig. 5b A sample of the core material (left) with the corresponding FTIR analysis results (right).



Fig.6 Clean metal surface versus unclean.

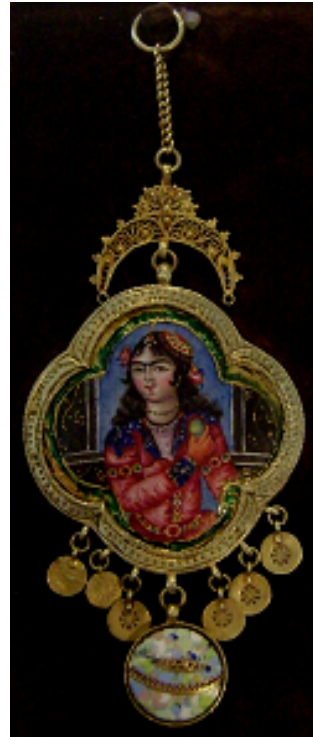


Fig. 7 The pendant after cleaning is complete.



Fig. 8 Plexiglass micro-environment showcase.

## التحليل الكيميائي والدراسة الفنية والصيانة لدلاية قجارية مموهة بالمينا التصويرية

د. وفاء أنور محمد ♦

### ملخص البحث :

يهدف البحث الى دراسة التركيب الكيميائي والخصائص الفنية الصناعية لدلاية قجارية مموهة بالمينا التصويرية. وقد استكملت الدراسة بأجراء عمليات التنظيف والصيانة و الأعداد للعرض المتحفي. وقد تطلبت عمليات الفحص و التحليل الكيميائي الدقيق استخدام الميكروسكوب الضوئي و الميكروسكوب الألكتروني الماسح المقترن بالتحليل الطيفي بالأشعة السينية، والتحليل بطيف الأشعة تحت الحمراء، والتحليل بتفلور الأشعة السينية. وقد أثبتت الدراسة أن الدلاية مصنوعة من شرائح الذهب الرقيقة و الأسلاك الذهبية مختلفة القطر ذات المقطع المستدير ونصف المستدير من عيارات متعددة كما تم التعرف على تركيب مادة المينا. وقد تناولت الدراسة تحديد وتحليل التقنيات الصناعية المختلفة التي طبقت لإنتاج هذه المشغولة مثل الدفع من الخلف (الريبوسية) و المحببات وأشغال السلك الدقيق (الشففتشي) و التحزيز والكبس بالأضافة الى معالجة السطح بالمينا المحجرة بالسلك (الكلوزونية) و المينا التصويرية. وقد أثبتت عمليات الفحص والدراسة أن الحلية الذهبية الرباعية لم تصنع من سبيكة مصممة من الذهب بل من الشرائح الذهبية الرقيقة المدعمة بحشو داخلي من الشمع. كما تم تصميم خزانة عرض مؤقتة للدلاية توفر درجة رطوبة نسبية ثابتة متحكم بها.

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