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Gilding on glass: New evidence from a 17th century flask found in Portugal

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ABSTRACT

In the present work, a set of archaeological glass fragments belonging to a singular 17th century glass flask with gilded decoration was sampled and analyzed. The fragments were excavated in Coimbra, Portugal. The main focus of this study is to bring to light the flask production technique, in particular the gilding process, and additionally, to investigate the object's provenance based on the composition of the glass. A comprehensive survey of ancient and traditional glass gilding techniques is also presented.

The glass and the gilded areas were characterized using micro energy dispersive X-ray fluorescence spectroscopy (μ-EDXRF), scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS), X-ray electron probe micro-analysis (EPMA) and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS).

The flask is made of soda-lime-silica glass and the use of coastal plant ashes is suggested by the relatively high content of MgO and K_2O, as well as by the presence of chlorine. The high alumina content excludes a Venetian provenance, and with regard to the contents of Na_2O, SiO_2, K_2O, and CaO, the glass composition is distinct from the *façon-de-Venise* glasses which have been studied, and only comparable to a few 17th century *millefiori* glasses, also recovered in Coimbra.

Lead was detected in the gilded areas suggesting that a lead based mordant was used in the gilding process to promote the adhesion of the gold leaf to the glass during the low temperature reheating of the object. The gold leaf composition was determined by LA-ICP-MS, and it was possible to ascertain its purity: between 22 and 23 gold carats.

1. Introduction

Archaeological glass with well-preserved gilded decoration is rather uncommon. In the present work, a comprehensive study of a 17th century gilded glass flask found in Portugal was carried out in order to, primarily, identify the gilding technique employed and, additionally, to investigate its provenance based on the composition of the glass. With this study, we aim to contribute to, and enrich, current knowledge on historical gilding techniques on glass, until now mostly known through recipes reported in historical treatises and from the making of replicas of ancient objects.

1.1. Gilding techniques on glass

Since ancient times, gold has been widely used in the decoration of glass vessels. Several studies have been carried out on the techniques used to attach gold to glass, combining the examination of preserved objects with the information provided by historical technical texts and the reproduction of ancient techniques (e.g. Charleston, 1972; Brill, 1991; Moretti and Toninato, 2001; Gudenrath, 2006; Verità, 2006; Rydlová and Drobny, 2007; Gueit et al., 2010).

Two main categories of gilding processes can be identified: i) early stage gilding: the process takes place during the blowing of the vessel; ii) post-formation gilding: the process takes place after the vessel is completely finished. The techniques that require the decorated glass to be covered with a protective glass layer (sandwich glass and *Zwischengoldglaser*: Kunckel, 1679: chapter XXVII, Moretti and Toninato, 2001, Whitehouse, 2007, Gudenrath, 2006, Rydlová and Drobny, 2007) are not considered in this study.

In early stage gilding, the decoration is obtained by picking up the gold leaf directly on the gather, forming the vessel, and heating it in

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the furnace while still attached to the blowpipe or pontil. This operation allows the gold to be embedded in the glass, thereby creating a durable attachment. Venetian glassmakers used this technique from at least the second half of the 15th century, and it is described in 16th century sources such as Biringuccio (1540) and Agricola (1556) (Gudenrath, 2006; Moretti, 2001).

In post-formation gilding, an already annealed glass object is decorated by means of gold paint or applied gold leaf. For painting, a gold suspension in a viscous liquid is prepared. Kunckel (1679, chapter XCIX) explains how to produce the so-called *shell gold*: this was a gold paint obtained by grinding gold leaf with sal-ammoniac (NH_4Cl) and gum Arabic. If gold leaf was used, a glutinous material, such as linseed oil, gum Arabic, egg white, varnish, Armenian bole (red clay), ochre (mostly ferric oxide, Fe_2O_3), or mastic was first spread on the glass with a brush. Then the gold leaf was applied over it and allowed to dry. When dry, the decoration was created by scraping away the gold leaf with a sharp tool (*sgraffito* technique) (Gasparetto, 1958; Moretti, 2001; Gudenrath, 2006).

Several texts dating from the 15th to 17th centuries, including a Portuguese one (Chagas (1767)) highlight the need for the use of size or mordant, obtained by adding to the above mentioned glutinous media a wide variety of compounds, most of them containing lead, such as cerussite ($PbCO_3$), minium (Pb_3O_4), litharge of silver (PbO with some silver), and lead glass. In addition, other substances such as borax, chalk ($CaCO_3$), ammoniac, cinnabar (HgS), *verdigris* (basic copper acetate, $Cu(OH)_2 \cdot (CH_3COO)_2 \cdot 5H_2O$), bones ashes (rich in Ca and P compounds), *coperosa* (zinc sulphate, $ZnSO_4$), rock alum (potassium aluminum sulphate $KAl(SO_4)_2 \cdot 12H_2O$) and vitriol (metallic sulphate) are also mentioned (*Dell'arte del vetro per musaico*, recipes no. LXXXVI and LXXVIII, in Milanesi, 1864; *Secreti diversi*, recipe no 339, in Merrifield, 1849, vol. 2; Moretti and Toninato, 2001, chapter LXXXIX; Kunckel, 1679: chapters XI–XXVI).

The need to reheat the gilded vessels is seldom referred to. Some recipes suggest that the gold foil can be attached simply using gum Arabic and reheating the object at a "moderate temperature" (for example Kunckel, 1679: chapters XI–XII). This seems to imply that the object is intended to be reheated at a temperature below the glass softening point, thus placing it in a muffle, instead of heating it in the furnace; in consequence, there is no need to reattach the object to the pontil. According to the description of the process made by Kunckel (1679: chapters XX–XXI), the mordant containing borax assures the best results because of the melting of the borax itself.

Nevertheless, the experiments carried out by W. Gudenrath have shown that if the glass is reheated to a temperature below its softening point, the gold applied with a simple glutinous medium is easily scraped off from the surface (author's experimentation at the Studio of The Corning Museum of Glass with W. Gudenrath, 2007). In order to permanently attach the gold to the surface of the glass, the vessel should be reattached to the pontil and carefully re-fired in the furnace until the walls have reached the softening point. The effectiveness of this method, mentioned by 16th century treatises for fixing enamels and used widely in Islamic and Venetian glass, was confirmed through experimental work carried out by W. Gudenrath (2006). As the objects need to be reattached to the pontil, most of the vessels gilded using this technique exhibit a distinctive double pontil mark.

1.2. Characteristics of gold and gold alloys circulating in Portugal during the 17th century

During the 17th and 18th centuries, in Portugal, gold was the major element of the opulent Baroque decorations, the so-called *talha dourada*, and was also widely used in polychrome wood sculptures, gold coins, jewellery and religious artefacts.

The gold from the coins struck in Portugal between 1483 and 1521 arrived from mines on the West coast of Africa, controlled by the Portuguese from 1482 and previously exploited by Islamic dynasties. In 1521–1523, a crisis in this region led to a decrease in the gold exported; however, these mines continued to be exploited by the Portuguese until 1638 (Guerra, 2008). From 1500, gold from East Africa reached Portugal as a tribute from the African king of the Quíloa kingdom (now Kilwa Kisiwani in Tanzania) to the Portuguese king D. Manuel I, and was used in the production of religious artefacts, such as the iconic Monstrance of Belém (Custódia de Belém) (Bastos and Franco, 2010). After 1530, the Portuguese were already established in the valley of the Zambezi River and controlling its gold mines, however, in Portugal there is no evidence that this gold had been used for minting coins (Guerra, 2008). Gold coins circulating in Portugal from 1521 to 1578, analyzed by Guerra (2008), seem to have been struck with a mixture of Colombian gold with West African gold. Nonetheless, East African gold may have reached Portugal and been used, for instance, in the manufacture of jewellery. This gold also reached India, trading with which was controlled by the Portuguese until 1629. Gold from other Latin America sources, namely Mexico and Peru, seem to have never reached Portugal (Guerra, 2008).

Several studies have been carried out focusing on the characterization of the gold leaf used in wood gilded ornaments (Moura et al., 2002; Serck-Dewaide et al., 2002; Bidarra et al., 2009; Le Gac et al., 2009), on the gold coins circulating in Portugal during this period (Guerra, 2008; Guerra and Calligaro, 2004) and one study focusing on a religious gold artefact (Oliveira and Ribeiro, 2010).

A survey of the results obtained is presented below, as it may contribute to the identification of the provenance of the gold used in the gilded flask.

During the Baroque period, in Portugal, gold leaf was beaten from high quality gold (23–24 carat) (Serck-Dewaide et al., 2002), or from alloys containing small amounts of Ag and/or Cu (18–23 carat) (Bidarra et al., 2009; Moura et al., 2002). The major elemental composition of gold leaf from different locations was reported by Moura *et al.* (2002): Au-Ag alloys were identified in the majority of the analyzed samples (Alcobaça, Azores, Aveiro and Coimbra); an Au-Ag-Cu alloy in Aveiro and Tibães; and finally an Au-Cu alloy in Tibães. The thickness of the gold leaf ranged from 1 to 2.5 μm in Coimbra, and from 3 to 4 μm in Azores (Serck-Dewaide et al., 2002).

The most iconic Portuguese religious gold artefact, the Monstrance of Belém, dating from 1506, was made with gold from Quíloa on the East African coast. Three different Au-Ag-Cu alloys were identified in this piece: alloy 1 (96–2–2 wt.%); alloy 2 (92–6–2 wt.%) and alloy 3 with Au between 84 and 88 wt.%, Ag between 10 and 14 wt.% and Cu ranging from 1 to 3 wt.% (Oliveira and Ribeiro, 2010).

The ratios Pt/Au and Pd/Au allow one to distinguish between the sources of the gold ores: the West African gold seems to have a lower Pt/Au ratio than the Colombian gold; the East African gold seems to be characterized by a higher Pd/Au ratio than the two aforementioned gold sources (Guerra, 2008; Guerra and Calligaro, 2004).

1.3. Archaeological context

Between 2000 and 2003 archaeological excavations were carried out in the Court of the University of Coimbra (Coimbra, Portugal), as part of a wider project involving the architectonic and artistic evaluation of the entire complex (Pimentel, 2005; Catarino and Filipe, 2006).

This investigation enhanced the historical knowledge of this area of town, which has been inhabited since the Roman *Aeminium* (the Roman name for Coimbra) till modern times. Of special interest were the remains of the fortification (*Alcácer*) belonging to the Islamic town of *Madinat Qulumbriya*. The building has a quadrangular plan, with towers leaning against the wall at regular intervals. The door, flanked by two towers, facing East, is today called *Porta Férrea*. It has always been the main access to the complex despite the alterations which took place after the Christian Reconquest of the town in 1064, when the building was first converted into the Royal Palace, and then, in 1537, chosen by King D. João III to host the University.