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Study of mosaic glass *tesserae* from Delos, Greece using a combination of portable μ-Raman and X-ray fluorescence spectrometry

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ABSTRACT

The present work discusses the results of an archaeometrical investigation on glass tesserae present in the mosaics from Delos (Greece) that was conducted *in situ* using two complementary non-invasive techniques, namely μ-Raman and portable X-ray Fluorescence spectrometry. The main objective of this work was to provide a compositional and technical characterisation of the glass tesserae. Since they are generally coloured and opaque or translucent, the study mainly focused on the identification of the employed colouring and opacifying agents.
The three hundred and fifty four mosaics from the Cycladic island of Delos (130 to 88 BCE) form one of the most relevant mosaic corpus of the Hellenistic period. These numerous mosaics are often of high quality and stand out by the large use of artificial materials, in particular glass and faience, which is remarkable for the period.
During three *in situ* analytical campaigns, around 270 glass tesserae were analysed by pXRF and nearly 40 tesserae using portable μ-Raman spectroscopy. This integrated approach allowed to gain an overview of the raw materials and the techniques employed in the production of these glasses. In particular, for all the tesserae the use of *natron* as a flux could be hypothesised. The majority of red glass tesserae were identified as a high-copper high-lead glass generally referred to as sealing-wax. The colourings agents used in the other tesserae (Fe, Co and Cu ions, $Pb_2Sb_2O_7$) are the common ones used in Antiquity and the antimony-based opacifiers are typical for the considered period and geographic area. Blue and turquoise tesserae remarkably show a highly variable lead concentration which appears correlated with antimony. This correlation raised the hypothesis that an antimony ore rich in lead was used for the opacification of these glasses.

1. Introduction

Mosaics are floor and wall decorations made of discontinuous elements which can be composed of various materials, both natural and artificial whose nature has changed over time. The earliest mosaics were produced using natural pebbles readily available from river-beds or from the seashore (Bruneau, 1976).

During the Hellenistic period, mosaicists began to integrate artificial materials (glass, faience and *terracotta*) to supplement the colours provided by stones in particular to reproduce the rich polychromy of contemporary paintings (Bruneau, 1987:55–86; Guimier-Sorbets and Nenna, 1992). In that period, the use of glass was mainly limited to the colours not easily available with natural stones, such as blue and green and more sporadically red and yellow.

From the mid first century CE onwards, glass tesserae became the most common material used for wall and vault mosaics (Sear, 1975) and remained seldom employed in floor ones.

Glass from the 800 BCE until 800 CE in the West and Middle East was mainly produced using a *natron*-based flux (Henderson, 1985) and a sand rich in calcium which is necessary to stabilize glass. Regarding the opacifiers, antimony-based compounds were employed since 1500 BCE and their use was predominant until the 4th century CE, calcium antimonate being a white opacifier and lead antimonate a yellow one.

This paper reports the results of an archaeometrical investigation on the glass tesserae of the mosaics from Delos (Greece) conducted *in situ* using two complementary portable and non-invasive techniques, namely portable X-ray Fluorescence Spectrometry and μ-Raman Spectroscopy. This work is part of a broader research program on vitreous materials employed in mosaics from Delos that constitutes the subject of the PhD thesis of the corresponding author. The main objective was to provide a compositional and technical characterisation of the glass tesserae. In particular, as they are coloured and generally opaque or translucent, special emphasis was put on the identification of colouring and opacifying agents. An important constraint was that the analytical techniques employed in the study of entire mosaics or fragments had

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to be strictly non-invasive (no sampling) and non-destructive (no damage). Evidently the application of portable non-invasive techniques on archaeological glasses without preliminary preparation has some limits which partly stem from the intrinsic features of the employed techniques. As for pXRF, light elements such as magnesium, aluminium, silicon or phosphorus are poorly quantified and sodium cannot be measured at all. Moreover, the measurement of other elements such as calcium might be deeply affected by the presence of surface weathering. As concerns portable μ-Raman spectroscopy, its non-confocal optics makes it sensitive to the spurious fluorescence that often hinders the spectra acquisition. Nevertheless, the merits of this kind of approach are mostly related to the possibility to analyse a large number of glass tesserae, providing statistically significant results on raw materials and production techniques.

1.1. Archaeological context

1.1.1. The mosaics of Delos

The mosaics of Delos are one of the most important corpus of mosaics produced during the Hellenistic period. Three hundred and fifty four pavements have been catalogued by Bruneau (1972), the vast majority dated between 130 and 88 BCE. Delos was declared free port in 167 BCE and developed into an important trade centre in the Mediterranean Sea, hosting a cosmopolitan population, and witnessed its greatest development till 88 BCE (Bruneau and Ducat, 2005: 31–48).

The mosaic pavements were made using a variety of techniques, such as chip and pebble mosaics, *tessellatum* and *vermiculatum*. *Tessellatum* refers to the use of generally square cut elements measuring more than 4 mm while *vermiculatum* conventionally refers to the use of smaller tesserae which can have various shapes (Bruneau, 1972: 32–35).

The decorated mosaics account to a hundred and twenty (Bruneau, 1972: 37) and represent a variety of decorative themes. In most cases, they were used to decorate private houses, but were also found in religious and public buildings.

A pioneer archaeological research on the use of vitreous materials in the mosaics from Delos has been conducted by Guimier-Sorbets and Nenna (1992, 1995). These publications give a thorough description of decor types, colours and techniques and point out the use of glass and faience. Vitreous materials were identified in nearly fifty mosaics. The great majority of glass elements were probably cut from glass rods (Guimier-Sorbets and Nenna, 1992: 626). In three pavements, the re-employment of opaque blue vessel fragments was reported (Bruneau, 1972: 245 and 256; Guimier-Sorbets and Nenna, 1992: 626).

Glass *tesserae* were mainly blue and green, less frequently red and yellow (Fig. 1). The use of violet glass tesserae was reported for two mosaics. As regards the chemical composition, only few glass tesserae have been analysed so far (Fiori, 1995).

1.1.2. Archaeological glass from Delos

The glass findings unearthed during the excavations conducted since 1873 by the French School of Athens were studied and classified by Nenna (1999). Around 1400 fragments of glass objects were discovered and most of them were dated from the end of the 2nd century to the early 1st century BCE. They include core-formed glasses, polychrome and monochrome moulded vessels and pieces of blown glass that were probably imported as finished products (Nenna, 1993, 1999). A large number of ornamental items (beads, pendants, elements mounted on rings, furniture inlays) were also collected. While no furnace, crucible and raw material deposits related to glassmaking were discovered on the site, numerous intermediate-products such as glass canes, rods along with some failed beads testify the existence of three workshops where glass was reworked in beads (Nenna, 1993, 1999).

Moreover another glass beads workshop was probably located near the Aphrodision of Stèsileôs. Indeed during recent excavations of this building, more then 6000 glass fragments have been discovered. Part of them is related to the occupation layers of the *oikoi* (4^{th}- early 1^{st} BCE) while the other consists of a glass bead workshop dump probably located near the Aphrodision which was reused as a backfill for the construction of the *oikos* 2 (Durvye et al., 2008). The latter contained more then 4000 glass fragments including different type of beads,

Fig. 1. Top left, turquoise and blue glass tesserae in a fragment of a three-strand guilloche (mosaic 336). Top right, green, light blue and dark blue in a meander (mosaic 210). Bottom left, yellow, red, blue, green and dark green glass tesserae in a meander (mosaic 229). Bottom right, detail of a vegetal garland (mosaic 68) showing the use of red glasses appearing in green due to weathering and green faiences.

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