ABSTRACT

The finding of considerable remains of glass mosaic floors, together with great stone mosaic floors dated back to Byzantine period (6th to 7th century A.D) from the Cross Church at the Jerash/Jerasa archaeological site in northern Jordan represented a strange phenomenon (glass mosaic). The chemical analysis of ancient glass mosaic tesserae can provide important information regarding the manufacturing technology of the glass mosaic made during a specific period. The aim of this study is to characterize the chemical and technological aspects of Byzantine glasses excavated from this main archaeological site. For this purpose, a considerable group of glass tesserae of different colours were collected and analyzed by XRF spectroscopy. XRD spectroscopy was used to identify the crystalline phases of glass tesserae and mineralogically to characterize the underlying lime mortar. All samples were investigated by optical microscopy. The results of chemical analyses indicated that the glass tesserae show a clear difference in chemical composition. The majority of the glass tesserae are compositionally homogeneous and belongs to the so-called natron-based silica–soda–lime glass type, whereas some samples are of relatively high level content of lead and aluminium. The tesserae colorants and opacifiers also are varied. Microscopic examination show obvious formal differences among glass tesserae samples.

KEYWORDS: Jerash; Byzantine mosaic; Glass tesserae; XRF; XRD.
1. INTRODUCTION

The modern village of Jerash, ancient Jerash during the Roman Empire's occupation of Jordan, is located 30 kilometers north of the capital Amman. During the Roman period, Jeresh was one of the major cites of the ancient world, as a member of the league of cities of the Decapolis (Browning, 1982; Lenzen et al., 1985) (Fig. 1).

Jerash is one of the most important archaeological sites in Jordan due to its prosperity in the first millennium AD including the Byzantine period. The excavation works were carried out in this important site by the British-American team from 1928 to 1934 (Crowfoot, 1930). From the latter period, sixteen churches and two chapels, most of which were with mosaic floors, were discovered.

Recent Archaeological excavations uncovered a church in October 2011 during the first season of excavation work carried out by the Institute for Mosaic Art and Restoration in Madaba (IMAR) directed by Dr. Adnan Shiyyab (Shiyyab, 2013). The newly discovered basilica called by the excavation manager "Cross church" that it consists of drawing of large cross (Figs. 2 and 3). The church is tentatively dated to the late 6th century A.D and early 7th century A.D., its walls were built with limestone blocks (Latitude: 32°16′50″ N; Longitude: 35°53′57″ E; Elevation above sea level: 528 m = 1732 ft).

Each wall consists of two rows and the space between them was filled with small stones, rubble, pebbles and a little of mortar. To the south and outside the church other archaeological features were found. These features are believed to be from a later phase and they include a rock-cut cistern connected with a channel of ceramic pipes.

A white mosaic floor was also uncovered south of the main building of the church. The floor is decorated with a repeated geometric shape, orthogonal patterns of adjacent scales, inside each one is a decoration of a rose in two colors, black and red. The frame of this mosaic floor was decorated a zigzag decoration between two straight lines (Figs. 3 and 4) (Shiyyab, 2013).
Glass tesserae were also found in the debris directly above the floor. Each piece consists of 40 small tesserae in different colors including green, black and blue still attached together with underlying mortar.

However the stone mosaic floor was randomly restored with glass tesserae which suggested that the Cross church experiences a dangerous kind of degradation: probably, the excavated glass mosaic tesserae was a part of wall mosaic before falling down to the floor (Fig. 5) (Shiyyab, 2013).

Glass has been used to produce tesserae for used in mosaic decoration for more than 2000 years, and became the most widely used material in mosaic decoration in Roman and Byzantine periods (4th-7th century A.D.). This is because their ability to reflect light as well as the range of color hues produced that could not be found in the nature (Farneti, 1993).

Tesserae glass material is obtained by a mixture of silica, alkali, lime and metal oxides as coloring agents. After the mixing these ingredients, the compound is fused in a furnace. It is then poured in the form of rod of variable diameter called "fillato rods" or dropped to the ground in the form of a cake, then the tesserae are obtained by cutting the cake using metal blade or light hammer.

When the glass became hard the so called "Tagliato" technique was used by place the glass cake on a chisel blade and then cut by hammer blow. A molten glass is poured onto a metallic table to obtain a round flat slab. This glass slab is subjected to a slow annealing process (to avoid the formation of stresses) and then cut to obtain many small square tesserae (Verità et al., 1996; James, 2006; Dal Bianco and Russo, 2012).

This work extends archaeometric knowledge of the glass mosaic tesserae of “Cross church at Jerash”, investigating the chemical composition and the nature of the glass matrix, colorants, and opacifying agents across a considerable set of mosaic tesserae of different colors.
2. MATERIAL AND EXPERIMENTAL METHODS

2.1 Analyzed samples

For experimental purposes, 15 glass tesserae of different colors (green, yellowish green, blue, turquoise blue, black, gray black, pale yellow and pale red) were selected. The set of samples is constituted by a piece of mosaic of 42 tesserae still attached to its original mortar, but unfortunately detached from the wall.

The originality of these tesserae is supported by their stylistic aspect and the technical considerations of the restorers. The sample was found during excavation work of the site in 2011.

Being already detached from the wall, there are no data on the original position of the sample itself apart from the fact that it belonged to the Cross church. Archaeologists suggested that glass tesserae are dated back to the Byzantine period (6th to 7th century A.D) (Shiyyab, 2013).

These samples have been used in this study not to cause further damages to the mosaics still in situ. To certain the analytical results, three glass vessel fragments found together with the glass tesserae in the same site were analyzed. Photographs of these glass tesserae are shown in Figures 6 and 7, where Table 1 summarises the visual description of these samples.

![Figure 6 The glass mosaics selected for experimental study.](image)

![Figure 7 Glass mosaic tesserae lifted from the underlying mortar](image)
Table 1 Description of the glass mosaic tesserae selected for experimental study

<table>
<thead>
<tr>
<th>Sample</th>
<th>Material</th>
<th>Colour</th>
<th>Opacity</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Glass tesserae</td>
<td>Green</td>
<td>Opaque</td>
</tr>
<tr>
<td>2</td>
<td>Glass tesserae</td>
<td>Green</td>
<td>Opaque</td>
</tr>
<tr>
<td>3</td>
<td>Glass tesserae</td>
<td>Yellowish green</td>
<td>Opaque</td>
</tr>
<tr>
<td>4</td>
<td>Glass tesserae</td>
<td>Yellowish green</td>
<td>Opaque</td>
</tr>
<tr>
<td>5</td>
<td>Glass tesserae</td>
<td>Dark blue</td>
<td>Opaque</td>
</tr>
<tr>
<td>6</td>
<td>Glass tesserae</td>
<td>Dark blue</td>
<td>Opaque</td>
</tr>
<tr>
<td>7</td>
<td>Glass tesserae</td>
<td>Turquoise blue</td>
<td>Opaque</td>
</tr>
<tr>
<td>8</td>
<td>Glass tesserae</td>
<td>Turquoise blue</td>
<td>Opaque</td>
</tr>
<tr>
<td>9</td>
<td>Glass tesserae</td>
<td>Black</td>
<td>Opaque</td>
</tr>
<tr>
<td>10</td>
<td>Glass tesserae</td>
<td>Black</td>
<td>Opaque</td>
</tr>
<tr>
<td>11</td>
<td>Glass tesserae</td>
<td>Gray black</td>
<td>Opaque</td>
</tr>
<tr>
<td>12</td>
<td>Glass tesserae</td>
<td>Gray black</td>
<td>Opaque</td>
</tr>
<tr>
<td>13</td>
<td>Glass tesserae</td>
<td>Pale yellow</td>
<td>Opaque</td>
</tr>
<tr>
<td>14</td>
<td>Glass tesserae</td>
<td>Pale yellow</td>
<td>Opaque</td>
</tr>
<tr>
<td>15</td>
<td>Glass tesserae</td>
<td>Pale red</td>
<td>Opaque</td>
</tr>
<tr>
<td>16</td>
<td>Glass tesserae</td>
<td>Pale red</td>
<td>Opaque</td>
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<tr>
<td>17</td>
<td>Glass vessel fragment</td>
<td>Yellowish green</td>
<td>Transparent</td>
</tr>
<tr>
<td>18</td>
<td>Glass vessel fragment</td>
<td>Yellowish green</td>
<td>Translucent</td>
</tr>
<tr>
<td>19</td>
<td>Glass vessel fragment</td>
<td>Yellowish green</td>
<td>Translucent</td>
</tr>
<tr>
<td>20</td>
<td>Mortar</td>
<td>White</td>
<td>-</td>
</tr>
</tbody>
</table>

2.2 Methods of analysis and investigation

A Bruker S4 Pioneer Wavelength Dispersive X-Ray Fluorescence Spectrometer (WDXRF) works under vacuum, voltage 20-60 KV, current 5-150 mA and a Power limit of 4050 watt, located at the Laboratories of the Natural Resources Authority of Jordan (NRAJ) was used to determine the chemical composition of the glass tesserae as well as vessel glass samples. This method was the most accurate method available for determining the elemental composition and the concentration of elements in the sample. This technique was preferred since it only required a small amount of the samples (Abd-Allah, 2011-2013).

For identifying the crystalline phases of glass tesserae and characterizing the underlying mortar, a X-Ray Powder Diffractometer Phillips [X Pert MPD] system with Cu Kα radiation (1.543 Ao) operating at reflection mode was used. For the powder method employed, the glass tesserae and mortar samples were perfectly cleaned from any dirt and deposits, finely powdered and prepared for analysis. An optical microscope (Nikon model H-III) was used for examining the textural characteristics of glass tesserae.

3. RESULTS AND DISCUSSION

3.1 XRF identification and characterization

The XRF results for the 15 glass tesserae and three glass fragments are reported in Table 2 and Figure 8. Regarding the glass tesserae, the major components of the raw glass vary in the following ranges: SiO₂ from 53% to 68%, Na₂O from 9% to 14%, CaO from 8% to 12%, and Al₂O₃ from 2.4% to 5.2%. The samples are characterized by low contents of potash (K₂O, 0.5-1.30%) and magnesia (MgO, 0.5-1.6%); hence, the raw material can be classified as natron based silica soda–lime glass (see the K₂O vs. MgO classification diagram of Figure 10). This type of raw glass is a peculiar feature of Roman and Byzantine glass finds from the Levant and Western Mediterranean area (Turner, 1956; Sayre and Smith, 1974; Henderson, 1985; Lirtizis et al., 1997; Freestone et al., 2002a, b; Arletti, 2005; Arletti et al., 2011; Abd-Allah, 2006, 2010, 2012), produced with siliceous-calcareous sands and characterized by low MgO and K₂O contents (less than 1.5%) and high Na₂O content, which usually ranges from 14% to 19%. In Europe, natron-like glass was widely used in glass production from the sixth and seventh centuries BC up to
the end of the first millennium AD, when it was substituted by plant ash. Moreover, the CaO vs. Al₂O₃ diagram (Fig. 9), which is largely used for discriminating the silica source, shows that the Roman-Byzantine tesserae mainly fall in the averages 8.7% and 3% for lime and alumina, respectively (Sayre and Smith, 1961; Lirtizis et al., 1997; Freestone et al., 2002b).

Among the few exceptions, samples 9 and 10 are characterized by a higher alumina content, 11 and 12 by higher lime contents, 13 and 14 by a lower lime and soda content. The samples that show very different ratios of the two components could be produced with a different starting raw material. The contents of chlorine (0.10%-0.65%) and sulfur (0.02%-0.05%) in the analyzed tesserae are derived by the presence of halite (NaCl) and thenardite (Na₂SO₄) in various proportions in the natron composition (Shortland et al., 2006; Abd-Allah, 2007; Henderson, 1985; Silvestri et al., 2008; Di Bella et al., 2013). Furthermore, the compositions of the three samples of vessel fragments indicate that the major components of these samples are: silica (SiO₂ avg. 68%), soda (Na₂O avg. 16.5%), lime (CaO avg. 8.9%) and alumina (Al₂O₃ avg. 2.4%).

Therefore, these glass tesserae are also of the soda-lime-silica (Na₂O-CaO-SiO₂) type, with low contents of potash (K₂O avg. 0.64%) and magnesia (MgO avg. 0.51%), the common type of ancient glass for more than three thousand years (Shortland et al., 2006; Abd-Allah, 2007; Henderson, 1985; Silvestri et al., 2008; Di Bella et al., 2013). This composition also corresponds to the previously defined Levantine I glass group, and is also typical of Byzantine and early Islamic glass from the Levant.

Furthermore, this composition revealed that the main raw materials from which these glass tesserae have been manufactured are Levantine coastal sand as a source of silica, natron as a source of alkali soda (used as flux), and lime (which is already present as impurity or shell fragments in the Levantine coastal sands) as a source of calcium (used as glass stabilizer) (Freestone et al., 2002a; Abd-Allah, 2010).

However it should be noticed that one final aspect to consider in more detail is the location of this material within what is known of trading patterns and cost in the Byzantine world. How easy, and likely, is it that glass or tesserae were transported across the empire, and how much might it all have cost? Written sources imply that the movement of tesserae around the Mediterranean was not unknown.

In the eighth century, mosaic tesserae and workmen appear to have been exported for the decoration of the Great Mosque in Damascus and the mosque at Medina. When mosaics were added to the Great Mosque in Córdoba, these tesserae too were said to have come from Byzantium (James, 2006).
Table 2 Compositions of the Byzantine glass mosaic tesserae and vessel glass selected for the analytical study obtained by XRF.

<table>
<thead>
<tr>
<th>Oxides</th>
<th>FeO</th>
<th>MnO</th>
<th>TiO₂</th>
<th>CaO</th>
<th>Na₂O</th>
<th>PbO</th>
<th>Total wt. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.60</td>
<td>0.09</td>
<td>0.08</td>
<td>8.20</td>
<td>1.08</td>
<td>0.21</td>
<td>16.60</td>
</tr>
<tr>
<td>2</td>
<td>0.67</td>
<td>0.16</td>
<td>0.09</td>
<td>8.21</td>
<td>1.08</td>
<td>0.21</td>
<td>16.60</td>
</tr>
<tr>
<td>3</td>
<td>0.53</td>
<td>0.30</td>
<td>0.08</td>
<td>8.10</td>
<td>1.04</td>
<td>0.20</td>
<td>16.60</td>
</tr>
<tr>
<td>4</td>
<td>0.69</td>
<td>0.09</td>
<td>0.08</td>
<td>8.00</td>
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<td>0.22</td>
<td>16.60</td>
</tr>
<tr>
<td>5</td>
<td>1.00</td>
<td>0.41</td>
<td>0.10</td>
<td>10.20</td>
<td>1.28</td>
<td>0.47</td>
<td>16.60</td>
</tr>
<tr>
<td>6</td>
<td>0.81</td>
<td>0.42</td>
<td>0.10</td>
<td>9.40</td>
<td>1.17</td>
<td>0.25</td>
<td>16.60</td>
</tr>
<tr>
<td>7</td>
<td>0.79</td>
<td>0.44</td>
<td>0.12</td>
<td>9.01</td>
<td>1.02</td>
<td>0.21</td>
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</tr>
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<td>0.40</td>
<td>0.10</td>
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<td>1.11</td>
<td>0.22</td>
<td>16.60</td>
</tr>
<tr>
<td>9</td>
<td>4.64</td>
<td>0.08</td>
<td>1.43</td>
<td>6.83</td>
<td>3.64</td>
<td>0.28</td>
<td>16.60</td>
</tr>
<tr>
<td>10</td>
<td>4.61</td>
<td>0.08</td>
<td>1.39</td>
<td>6.22</td>
<td>3.59</td>
<td>0.26</td>
<td>16.60</td>
</tr>
<tr>
<td>11</td>
<td>1.47</td>
<td>0.98</td>
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<td>12.22</td>
<td>0.92</td>
<td>2.39</td>
<td>16.60</td>
</tr>
<tr>
<td>12</td>
<td>1.46</td>
<td>0.96</td>
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<td>12.00</td>
<td>0.94</td>
<td>2.41</td>
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</tr>
<tr>
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<td>14</td>
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<td>5.72</td>
<td>0.75</td>
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</tr>
<tr>
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<td>0.25</td>
<td>0.08</td>
<td>8.00</td>
<td>1.07</td>
<td>0.22</td>
<td>16.60</td>
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<tr>
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<td>0.26</td>
<td>0.08</td>
<td>8.02</td>
<td>1.05</td>
<td>0.20</td>
<td>16.60</td>
</tr>
<tr>
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<td>0.56</td>
<td>0.02</td>
<td>0.09</td>
<td>10.00</td>
<td>0.96</td>
<td>0.15</td>
<td>16.60</td>
</tr>
<tr>
<td>18</td>
<td>0.60</td>
<td>0.02</td>
<td>0.08</td>
<td>8.60</td>
<td>0.65</td>
<td>0.09</td>
<td>16.60</td>
</tr>
<tr>
<td>19</td>
<td>0.75</td>
<td>0.02</td>
<td>0.11</td>
<td>8.40</td>
<td>0.58</td>
<td>0.04</td>
<td>16.60</td>
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<td>20</td>
<td>0.36</td>
<td>0.01</td>
<td>0.02</td>
<td>5.10</td>
<td>0.03</td>
<td>0.04</td>
<td>16.60</td>
</tr>
</tbody>
</table>

3.2 Glass colorants and opacifiers determination

According to Fig. 4 the glass tesserae are of different colors (Green, yellowish green, blue, turquoise blue, black, gray black, pale yellow and pale red) and opaque. It was stated that the color of glass is due to the presence of coloring transition elements in the glass batch, most of them are recorded in XRF results as shown in Table 2.

Manganese (MnO,0.04-0.98%) was present as minor component in the original sands or intentionally added to color the glass tesserae purple or deep blue. In particular, in some purple tesserae manganese was intentionally added to obtain the desired color nuances. On the contrary, TiO₂ present in 0.7% -1.43%, and hence it is ascribable to be intentionally added as glass opacifier rather than to be impurities of heavy minerals in the raw sands. As regards antimony oxide, largely used as decolorizing and opacifying agent, it is particularly abundant in the opaque blue and in the white tesserae. Lead oxide contents are very high in the opaque yellow and red tesserae (16-18%) and relatively high in the green and yellowish green ones (20-35%). The relatively high concentration of PbO suggests the addition of lead as a melting agent and to improve the brilliance and chromatic aspect of the tesserae beside its role as opacifying agent of glass. Except the black tesserae samples 9 and 10, iron oxide (FeO) is present as an impurity associated with sands, and it is almost exclusively responsible for colouring glass to light green and yellowish green (less than 1%), whereas in some of a set samples it possibly intentionally added to color the glass black or blue (exceed 1%). The relatively high content of copper oxide (0.20-0.72%) indicate that it was used as colorant agent to color the glass tesserae blue, green, yellow and black (Di Bella et al., 2013; Jackson, 2005; Mirti et al., 2009; Abd-Allah, 2009; Croveri et al., 2010).
In addition to the high copper content, the red tesserae are also characterized by high concentrations of lead. FeO content, in particular, is the highest among the studied sample 11 and 12 (FeO-1.47 %), suggesting the deliberate addition of Fe bearing materials to the melt, with the aim of preventing copper oxidation (Croveri et al., 2010; Arletti et al., 2011). The relatively high concentration of PbO (about 2-3%) suggests the addition of lead as a melting agent and to improve the brilliance and chromatic aspect of the tesserae.

3.3 XRD crystalline phases identification

X-ray diffraction investigations of the opaque tesserae allowed the identification of colorant/opacifier phases, such as (A) metallic Cu+, forming clusters of different dimensions inside the red tesserae; (B) lead pyroantimonate (PbSb2O7) in the yellow and green tesserae, which is corresponding to the mineral phase bindheimite. Bindheimite is one of the most widely used yellow pigment in the technological history of glass production (Di Bella et al., 2013; Shortland, 2002, 2005) and has been already found by Croveri et al. (2010) and Di Bella et al. (2013) in Roman glass mosaics tesserae from Italy; and (C) both the hexagonal CaSb2O6 and the orthorhombic Ca2Sb2O7 calcium antimonate phases in the opaque blue, turquoise, and black tesserae. These opacifying agents are present in the glass network as modifiers rather than network formers (Newton and Davison, 1989). Regarding the underlying mortar, the results of the selected samples shown in Table 3 indicate that they are mainly composed of the calcium carbonate mineral calcite (CaCO3), and quartz (SiO2) as a minor component. These results are matches that obtained by XRF analysis where the percentage contents of CaO and SiO2 were 55.10% and 0.89% respectively, which means that the mortar used in the construction of the floor is a localized Lime mortar (Abd-Allah, 2011).

Table 3 Mineralogical compositions of the mosaic tesserae and underlying mortar samples obtained by XRD.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Colour</th>
<th>Crystalline phases</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Green</td>
<td>Lead pyroantimonate (PbSb2O7)</td>
</tr>
<tr>
<td>5</td>
<td>Blue</td>
<td>Hexagonal CaSb2O6 (Calcium antimonite)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Orthorhombic Ca2Sb2O7 (Calcium antimonite)</td>
</tr>
<tr>
<td>7</td>
<td>Turquoise blue</td>
<td>Hexagonal CaSb2O6 (Calcium antimonite)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Orthorhombic Ca2Sb2O7 (Calcium antimonite)</td>
</tr>
<tr>
<td>10</td>
<td>Black</td>
<td>Hexagonal CaSb2O6 (Calcium antimonite)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Orthorhombic Ca2Sb2O7 (Calcium antimonite)</td>
</tr>
<tr>
<td>13</td>
<td>Pale yellow</td>
<td>Lead pyroantimonate (PbSb2O7)</td>
</tr>
<tr>
<td>15</td>
<td>Pale red</td>
<td>Metallic Cu+, copper oxide</td>
</tr>
<tr>
<td>20</td>
<td>Mortar</td>
<td>Calcite , Calcium carbonate (CaCO3), Major</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Quartz, Silicone oxide (SiO2), Trace</td>
</tr>
</tbody>
</table>

3.4 Optical microscopy observation

Micro images (2.5-10X) of glass tesserae obtained by optical microscopy (Fig. 10) show that the texture of the green glass tesserae, has very homogeneous textures, without inclusions. The red, pale yellow, yellowish green and turquoise blue studied tesserae are quite homogeneous, with the presence of small bubbles and micro pits. The black and dark blue glass tesserae exhibit a spongy texture appearance with inclusions and the presence of diffused deformed bubbles and large-size pits. These features are certainly due to the defects of glass batch manufacture related to the low melting point of glass raw materials (Newton and Davison 1989; Abd-Allah, 2012, 2013).
4. CONCLUSION

A multi-technique investigation, based on different experimental methods (XRF, XRD, and OM), was conducted to study characterize the chemical composition, mineralogical structure and texture morphology of a considerable suite of Byzantine glass mosaic tesserae from the Cross church in Jerash archaeological site in northern Jordan. The result of this study contribute to widen our knowledge on the ancient materials and technological processes applied for the production of the glass tesserae of one of the most important examples of Roman and Byzantine mosaic art in the Mediterranean area. Chemical data indicate that the raw glass used to produce the tesserae is compositionally homogeneous and belongs to the so-called natron-based silica–soda–lime glass type (Liritzis et al., 1997).

The results on major, minor, and trace elements suggest that the raw glass, similar to that used for other glass objects of common use (like beakers, bottles, cups, etc.) produced from the 4th to 7th century AD, could have been imported from one of the few large production centers located in the Mediterranean Basin. X-ray diffraction investigations of the colored and opaque tesserae allowed the identification of colorant/opacifier phases, such as metallic Cu+, forming clusters of different dimensions inside the red tesserae; lead pyroantimonate (Pb₅Sb₄O₁₁) in the yellow and green tesserae; and both the hexagonal (CaSb₂O₆) and the orthorhombic (Ca₂Sb₂O₇) calcium antimonate phases in the opaque blue, turquoise, and black tesserae. Furthermore the type of underlying mortar was characterized as a localized lime mortar. Microscopic examination of glass tesserae texture allowed the detection of the formation of diffused deformed bubbles, large-size pits, and spongy texture appearance with inclusions, which are certainly due to the defects of glass batch manufacture related to the low melting point of glass raw materials.

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