ABSTRACT

The aim of this research is to study the deterioration resulting from burial context on archaeological glass. Investigations were performed on a series of Islamic glass fragments coming from different excavation sites (Deir El-Ghannam and Deir El-Banat) in Fayoum which was a major manufacturing place for archaeological glass during Islamic periods. The mineralogical and elemental compositions of glass fragments and soil were determined by Energy dispersive X-ray (EDX) methods, while glass fragments’ surfaces were examined by Scanning Electron Microscope (SEM) and USB Digital Microscope. Deterioration aspects resulting from being buried varied among excavations in the Fayoum, according to chemical composition and soil moisture’s proportion which increase in Deir El-Ghannam and decrease in Deir El-Banat. The study shows that different kinds of salts “Chloride and Sulphate”, dirty layers, soil deposits affect the thickness and colour of glass, on top of glass corrosion layers. This study resulted in the conception of a methodology to treat the deterioration aspects.

KEYWORDS
Deterioration, Burial environment, Excavation, Deir El-Banat, Deir El-Ghannam

1. INTRODUCTION

Fayoum was a major place for manufacturing archaeological glass during Islamic periods. Despite that this production has not been comprehensively examined. By focusing on the material coming from the excavations of two monasteries, this study aims to identify and analyse the chemical composition, soil moisture content, and other soil properties to
determine the impact of burial environment on glass fragments from the sites of Deir El-Ghannam and Deir El-Banat in Fayoum.

The monastery of Deir El-Ghannam is located between the monasteries of Gabriel and of St. Anba Abram in Azab, Fayoum, while Deir El-Banat is about one kilometre northeast from the monastery Gabriel. There are excavations belonging to the Antiquities Authority.¹

Glass is a comparatively durable material when buried.² The degradation of buried glass depends on water corrosion. The leaching of the components of glass and an ion exchange reaction between the glass and the soil are accompanied by the migration of water into the alteration layer. In addition, corroding products on the surface may contain water within their crystalline structure.³

However, corroded glass surface occurs in wet soils and moist leading to a transparency loss and the formation of a surface crust rich in silica but exhausted of its basic ions. The glass is weakened by this process which may accelerate the shattering of thinner objects.⁴

![Figure 1 Location of Deir El-Ghannam and Deir El-Banat](image)

The surface degradation rate in soil is affected by the composition of glass and not easily expected.⁵ The alkali content and type are critical. In general ancient glass is more resistant to chemical attacks than the medieval one as wood ash containing potassium (K) started replacing soda ash in its manufacture. Under moderately alkaline and acidic conditions (pHb9), alkali ions are leached from the glass matrix; under more alkaline conditions, hydroxyl ions disrupt silicon–oxygen bonds within the structure of the silica.⁶

Layers of laminar surface are easily formed in situations of higher PH alkaline;⁷ these layers may be iridescent. In the driest soils, coatings of surface and other decoration on glass are expected to decompose very rapidly beyond 100 year (b100 y). The strong dependence of glass corrosion rates on manufacture and material composition leads to

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¹ Andraos, “Coptic History”, p. 59.
³ Roemich et al., “Archaeological Glass”, pp 137-149
⁵ Van Giffen et al., “Weathered Archaeological Glass”
⁷ Roemich et al., “Results from burial experiments”, pp. 97-108.
doubt any expectation of degradation of the surface relative to the different kinds of soil. In arid soils, corrosion is less expected; soils which are highly alkaline, are the most corrosive.

While glass is well preserved in soil, it tends to shatter and the resulting shards may be dispersed. Physical damage to glass buried in soil can be due from static and dynamic forces. Static forces increase from the treading action of people, animals and vehicle movements may propagate in the subsoil. In soils where clay minerals are present, these will potentially create destructive mechanical forces during wetting-drying cycles. Soil stiffness, which is a measure of resistance to deformation, will affect the likelihood that brittle objects will be fractured. For instance, a dry clay soil will be more resistant to deformation and better protect objects from shattering than a wet sandy soil. However, within most soils, glass is highly resistant to degradation and will be well preserved. The exception lies in damp and strongly alkaline soils.

Glass and weathering crusts become obscured through insoluble salts’ encrustations. These are prevalent where the excess lime in glass is leached out, to be deposited as a whitish deposit on the surface or within the decayed layers of glass.

2. MATERIALS AND METHODS

2.1 Samples

2.1.1 Glass Samples

Two glass samples were selected from both sites of Deir El-Banat and Deir El-Ghannam, the excavation sites of which are shown in Figures 2 and 3. Figures 4 and 5 display the examination of the glass samples.

2.1.2 Soil Samples

Two samples from burial environment “one from each site”. Figures 7 and 8 show an analysis of the soil samples from Deir El-Banat and Deir El-Ghannam.

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8 Dain-Owens et al., “The risk of harm to archaeological artefacts”, pp. 1175-1186
9 Mark et al., “Predicting the preservation of cultural artefacts” pp. 249-263.
10 Cronyn, “The Elements of Archaeological Conservation”
2.2  **USB Digital Microscope**
The glass samples were examined by Leuchtturm USB Digital Microscope (China) with 20 to 500x zoom, 8 LED lights with Measurement Software.

2.3  **SEM-EDX**
Scanning Electron Microscope (SEM) images of the glass samples were taken using a Jeol (Tokyo, Japan) JSM 5600 LV equipped with an Oxford Instruments 6587 EDX microanalysis detector. The images were taken under low vacuum conditions where samples did not show any charging effects. Energy Dispersive X-ray microanalysis (EDX) was used to obtain information on the elemental composition of the excavation soil and glass samples.

2.4  **Soil Moisture Content (oven-drying method)**
Samples of soil from both areas were taken to calculate the moisture content (Table 1) according to the following equation:

\[
\text{Wet soil weight} = W_2 - W_3 \\
\text{Dry soil weight} = W_3 - W_1
\]

The percentage of the wet / dry soil = W.C% = \(\frac{(W_2 - W_3)}{(W_3 - W_1)} \times 100\)

Where: W1 is “weight of empty container”, W2 is “Container + wet soil”, and W3 is “Container + dry soil”.

The dry soil was obtained by being exposed to an oven at 105°C for 24 h.

3.  **RESULTS**
3.1  **USB Digital Microscope Examination**
The examination by USB Digital Microscope for Deir El-Ghannam samples A& B shows glass corrosion layers, glass dulling and iridescence (Figure 4).

![USB Digital Microscope photos for Samples A and B from Deir El-Ghannam](image-url)
The photographs done by USB Digital Microscope on the Samples C and D from Deir-El-Banat excavation show soil deposits, dirty layers, salt crystals, brown spots, and air bubbles inside the glass material (Figure 5).

![USB Digital Microscope photos for Samples C and D from Deir-El-Banat excavation](image)

Figure 5 USB Digital Microscope photos for Samples C and D from Deir-El-Banat excavation

### 3.2 SEM- EDX

The examination by SEM clearly shows glass corrosion, gaps, cracks and salt crystals in Deir El-Ghannam.

SEM photos of Samples A and B from Deir El-Ghannam display glass corrosion layers, salt crystals and gaps (Figure 6).

![SEM photos for Samples A and B and D from Deir El-Ghannam excavation](image)

Figure 6 SEM photos for Samples A and B and D from Deir El-Ghannam excavation
The results of EDX microanalysis indicate that the components of the corroded glass Sample A of Deir El-Ghannam excavations show the following major components: Silica (SiO$_2$ 71.13%), Soda (Na$_2$O 7.85%), Potash (K$_2$O 138%), Lime (CaO 11.88%), Alumina (Al$_2$O$_3$ 4.69%) and a small percentage of Chlorine (Cl$_2$O 1.67%) (Figure 7).

Figure 7 The components of Sample A from Deir El-Ghannam excavation by EDX

The EDX of Sample B corroded glass (Figure 8) indicates that the major components are: Silica (SiO$_2$ 71.73%), Soda (Na$_2$O 2.62%), Potash (K$_2$O 1.24%), Lime (CaO 6.36%), Alumina (Al$_2$O$_3$ 7.77%), Chlorine (Cl$_2$O 1.90%), Magnesium oxide (MgO 2.53%) and finally Iron oxide (Fe$_2$O$_3$ 5.84%).

Figure 8 The components of Sample B from Deir El-Ghannam excavation by EDX

Samples C and D from Deir El-Banat are in good condition. SEM shows dirty layers and soil deposits (Figure 9).
Figure 9 SEM photos for Samples C and D from Deir El-Banat excavation

The EDX Microanalysis for Sample C from Deir-El-Banat shows the following components: Silica (SiO$_2$ 65.62%), Soda (Na$_2$O 5.33%), Potash (K$_2$O 2.41%) Lime (CaO 12.18%), Alumina (Al$_2$O$_3$ 8.27%), Chlorine (Cl$_2$O 2.28%), Magnesium oxide (MgO 2.24%), and Iron oxide (Fe$_2$O$_3$ 5.23%) (Figure 10).

Glass sample D indicates that the major components are: Silica (SiO$_2$ 66.63%), Soda (Na$_2$O 1.88%), Potash (K$_2$O 2.28%), Lime (CaO 9.23%), Alumina (Al$_2$O$_3$ 5.69%), Chlorine (Cl$_2$O 3.12%), Magnesia (MgO 2.41%), and Sulphur trioxide (So$_3$ 3.24%) (Figure 11).
3.3 Soil analysis

3.3.1 EDX

The results of EDX Microanalysis indicate that the components of the soil sample of Deir El-Ghannam are: Silica (SiO₂ ~ 49.95%), Lime (CaO ~ 12.50%), Alumina (Al₂O₃ ~ 13.18%), Magnesium Oxide (MgO ~ 2.77%), Soda (Na₂O ~ 3.77%), Potash (K₂O ~ 1.46%), Chlorine (Cl₂O ~ 2.67%), Iron Oxide (Fe₂O₃ ~ 9.25%), and finally Sulphur trioxide (So₃ 3.03%) (Figure 12).

The components of the sample of soil from Deir El-Banat are: Silica (SiO₂ ~ 62.57%), Lime (CaO ~ 8.05%), Soda (Na₂O ~ 2.44%), Alumina (Al₂O₃ ~ 4.13%), Potash (K₂O ~ 2.60%), Chlorine (Cl₂O ~ 1.96%), Carbon oxide (Co₂ ~ 14.32%), and Sulphur trioxide (So₃ 3.91%) (Figure 13).
3.4 **Soil Moisture Content (oven-drying method)**

The results showed different moisture contents between Deir El-Ghannam and Deir El-Banat soils (Table 1).

<table>
<thead>
<tr>
<th>Samples No</th>
<th>Container weight (gm)</th>
<th>Container + wet soil (gm)</th>
<th>Container + dry soil (gm)</th>
<th>Dry soil (gm)</th>
<th>Moisture content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>24.2</td>
<td>55.6</td>
<td>52.3</td>
<td>29</td>
<td>28.80%</td>
</tr>
<tr>
<td>2</td>
<td>24.2</td>
<td>54.8</td>
<td>52.3</td>
<td>28.1</td>
<td>8.90%</td>
</tr>
<tr>
<td>3</td>
<td>24.2</td>
<td>55.3</td>
<td>52.4</td>
<td>28.2</td>
<td>10.28%</td>
</tr>
<tr>
<td>4</td>
<td>24.2</td>
<td>55.4</td>
<td>54.6</td>
<td>3.4</td>
<td>2.63%</td>
</tr>
<tr>
<td>5</td>
<td>24.2</td>
<td>55.1</td>
<td>54.2</td>
<td>30</td>
<td>3.00%</td>
</tr>
<tr>
<td>6</td>
<td>24.2</td>
<td>54.2</td>
<td>53.1</td>
<td>28.9</td>
<td>3.81%</td>
</tr>
</tbody>
</table>

4. **Discussion**

Deir El-Ghannam glass samples suffer from different deterioration phenomena, such as “hydrogen glass and Corrosion layers which look like gel layers, where it causes and increases the degree of glass dulling.”¹¹ Hydrogen glass contains to ion exchange’s reaction between the glass surface and water.¹² &¹³ In addition, iridescence (play of colours) appears clearly on the surface of Deir El-Ghannam glass Sample B in a thick

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¹¹ Pollard and Heron, “Archaeological chemistry” p 11-25
¹² Ryan et al., “Glass Deterioration”, pp.839-844
¹³ Bates et al., “Experimental Hydration Studies”, p. 123
layer when light is reflected. This phenomenon expresses the high dangers rate for damaged glass.\textsuperscript{14}

Glass corrosion for Samples A and B can be classified according to the corrosion ratio to corrosion with non-noble or malignant patina heterogeneous, layer-corrosion.\textsuperscript{15} SEM confirmed the appearance of glass corrosion through its distinctive shape which looks like a beehive.\textsuperscript{16}

Deir El-Banat glass samples were in a good condition containing, just dirty layers, soil deposits, and salt crystals in addition to air bubbles which happened during the manufacturing process.\textsuperscript{17} There were brown spots inside the glass of “Sample D” due to iron oxide being used in the manufacturing process.\textsuperscript{18}

All glass samples in Deir El-Ghannam and Deir El-Banat can be classified as (Soda-Lime-Silica) glass, the type of ancient glass popular for more than three thousand years.\textsuperscript{19\&20} This composition reveals that the main raw materials from which these raw glass fragments were, sand as a source of silica, natron as a source of alkali soda and finally lime as a source of calcium.\textsuperscript{21}

There was a high percentage of silica in the “corroded glass” samples of Deir El-Ghannam due to the glass component solution and deposition of silicon ions on the surface in the soil (glass corrosion layers). In addition to the low percentage of alkaline soda and potash as a result of the same reason which came from the effect of burial environment.\textsuperscript{22} The low content of potash and magnesia indicates that this is natron-based glass.\textsuperscript{23}

There was chlorine in all samples as the burial environment consisted of halite (NaCl).\textsuperscript{24} The amount of sulphur oxides and chlorine in the glass samples of Deir El-Banat is due to natron, which contains thermadite (Na₂SO₄) and halite (NaCl).\textsuperscript{25} Iron oxide is an impurity associated with sand, almost exclusively responsible for colouring glass a yellowish light green, the same colour than the Samples C and D.\textsuperscript{26 27}

EDX analysis for Deir El-Ghannam soil revealed that the soil is a wet and sandy one, rich in lime, alumina, and iron oxide. In addition, there is an increasing amount of moisture content which causes the glass corrosion.\textsuperscript{28\&29}

\begin{itemize}
\item \textsuperscript{14} Davison, “Caring for antiquities”, p 48-52
\item \textsuperscript{15} Abd-Allah, “Study of the effective factor on deterioration of buried glass” pp. 153-154
\item \textsuperscript{16} Hamad, “Study of factors affecting deterioration of archaeological glass”, pp. 140-143
\item \textsuperscript{17} Dawi, “Restoration and conservation of archaeological glass in Egypt”, pp. 93-101
\item \textsuperscript{18} Hamad, “Restoration and conservation of a model of archaeological glass”, pp.51-58
\item \textsuperscript{19} Tite et al., “The composition”, pp. 1284-1292
\item \textsuperscript{20} Degryse et al., “A geochemical study of Roman to early Byzantine Glass”, pp. 287-299
\item \textsuperscript{21} Abd-Allah, “Chemical characterisation”, pp. 1866-1874
\item \textsuperscript{22} Abd-Allah, “Study of the effective factor”, pp. 98-105
\item \textsuperscript{23} Silvestri et al., “The colourless glass of Julia Felix”, pp.331-341
\item \textsuperscript{24} Hamad, “Study of factors affecting deterioration of archaeological glass”, p.143
\item \textsuperscript{25} Abd-Allah, “Devitrification behavior of corroded”, pp. 39-49
\item \textsuperscript{26} Jackson, “Making colourless glass”, PP. 763-780
\item \textsuperscript{27} Hamad et al., “Conservation and protection”
\item \textsuperscript{28} Seas., “A conservation manual for the field archaeologist” , PP. 45-64
\end{itemize}
On the other hand, Deir El-Banat soil classified as dry and sandy soil, according to its high percentage of silica and low moisture content, which matches the good level of preservation for its archaeological glass.\(^{30}\)

**Methodology plan to treat the deterioration aspects**

Using Calcium acetate \((\text{CH}_3\text{COO})_2\text{Ca}\) and Sodium acetate \((\text{CH}_3\text{COO})\text{Na}\) as a substitute for Calcium and Sodium ions, in addition to Ethyl silicate to consolidate corroded glass.\(^ {31}\)

Dirty layers and soil deposits should be removed carefully by mechanical wet cleaning with distilled water.\(^ {32}\) It is necessary to use the Japanese tissue paper moistened with distilled water to extract soluble salts. After that, it is possible to strengthen and isolate the archaeological weak glass using paraloid B72 dissolved in acetone 3 % + ethyl silicate dissolved in 5 % alcohol.\(^ {33}\)

**CONCLUSIONS**

Deterioration components as a result of the chosen burial environment are glass corrosion layers differ in their thickness and colours, Different kinds of salts “Chloride and Sulphate”, Dirty layers, Soil deposits, and Cracks.

Fayoum excavations held in Deir El-Ghannam and Deir El-Banat were made in sandy soil rich in salts and iron oxides. Deterioration aspects which result of being in a burial environment, were different in Deir El-Ghannam and in Deir El-Banat, according to soil chemical composition, and soil moisture content. Indeed, the latter is higher in Deir El-Ghannam than in Deir El-Banat.

The archaeological glass from Deir El-Ghannam suffered from the high moisture content, which caused over time corrosion layers on the surface of the glass. It brought salts inside the glass layers and induced crystallisation of the salts after the excavation, which is not seen in Deir El-Banat samples.

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\(^{29}\) Shelby., “Introduction to glass science”, PP. 18-39  
\(^{30}\) Plenderleith et al., “Results from burial experiments”, PP.97-108  
\(^{31}\) Abd-Allah., “Study of the effective factor”, P.195  
\(^{32}\) Ling, “Conservation of Hellenistic vessel glass”  
\(^{33}\) Hasan et al., “An experimental study”, pp. 48-53
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