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DAMAGE ASSESSMENT AND CHEMICAL CHARACTERIZATION OF THE ARCHAEOLOGICAL GLASS WEIGHTS KEPT AT RASHID STORE OF ANTIQUITIES: A CASE STUDY

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Abstract:
The present research paper presents a series of glass weights dated back to the Fatimid Era (4th H.-10th G. century) from the excavations of Abu Mina in Alexandria. These weights have been kept in the Rashid store of antiquities since 1995 and suffered from many aspects of deterioration. The paper primarily aims to assess the deterioration state of glass weights according to the storage environment and diagnose the various phenomenon of decay. The glass weights understudy were examined by the USB digital microscope and the Scanning Electron Microscope (SEM) and characterized chemically using EDX. Furthermore, temperature, relative humidity, and light were monitored at the Rashid store of antiquities during summer and winter to determine their impacts on the degree of damage to the glass weights understudy. The microscopic investigation revealed the formation of weathering crusts, cracking, pitting, and iridescence layers on the surface of glass weights. SEM- EDX showed that those glass weights are entirely corroded. It also showed that those glass weights are of the soda-lime-silica type. Monitoring relative humidity, temperature, and light at the Rashid store of antiquities showed that the proportions are higher than the permissible ratios for preserving the glass antiquities, playing a clear role in exposing glass weights to intensive deterioration.

1. Introduction

Glass weights are one of the most important archaeological documents in bridging the gaps of the dynasties. They show the names and titles of caliphs and sultans during whose reign they were made. The study of their inscriptions also shows the developments of the shapes of letters, types of lines, and accuracy in drawing decorations and writings [1-3]. Glass weights show the sophistication of the glass industry and colors. They were used to maintain the weight from manipulation and cheating. They were intended to weigh dinars, dirhams, precious jewels, and drugs to maintain weight stability. Glass weights were preferred because it was noted that they were less likely to corrode in a relatively short time compared to metal coins, relatively inexpensive, easily detectable if tampered with, and produced by stamping process [4,5]. The earliest introduction of glass weights in the Islamic era was during the reign of the Umayyad caliph Abdul Malik Ibn Marwan. Then, they were developed during the Abbasid, Tulunid, and Fatimid periods [6]. In the early Fatimid era, glass weights were free of any indication of weight or value of the currency. They got worse at the time of the Ayyubid and Mamluk eras [7]. It is well-known that glass composition involves (silica) as the main component, usually referred to as the glass former. Adding soda and potash, the fluxing mat-erials, to silica- lowers the melting point of the mixture and alters the viscosity of the molten glass- are known as the glass modifiers lime, which stabilizes and imp-roves the chemical durability of the glass [8.9]. Glass is exposed to damage if it is preserved in an unsuitable storage envir-onment. The alteration of glass in storage environments is a very complex process due to numerous factors. It depends both on the characteristics of the glass (e.g., composition, heat treatment, and surface roughness) and the external conditions, such as relative humidity, temperature, and air pollution [10]. It also depends on the use of unsuitable storage materials, such as wooden boxes, which emit organic acids, e.g., acetic acid, formic, and formaldehyde, that damage the unstable glass [11]. The chemical decay of the glass often starts when its alkaline, soda, potash, or lime components are leached by water from the surrounding environment (leaching is the process of the extraction of the soluble co-mponents of a solid by their dissolution, usually in water but in mild acids, as well [12]. Two dominant mechanisms, i.e., ion exchange and network dissolution, produce degradation layers with a different com-position from the bulk glass. Concerning water adsorption at the surface, three com-peting simultaneous reactions result in the hydration of the glass [13,14], they are:

1) **Ion-exchange**: When cations, such as alkali ions, are replaced by H^+ or H_3O^+ $\equiv Si\text{-}O\text{-}\ Na^+ + H^+ \rightarrow \equiv Si\text{-}OH + Na$ $\equiv Si\text{-}O\text{-}\ Na^+ + H_3O^+ \rightarrow \equiv Si\text{-}OH + H_2O + Na$
2) **Hydration**: When the molecular water enters the glass as an intact solvent $\equiv Si\text{-}O\text{-}\ Na^+ + H_2O \rightarrow \equiv Si\text{-}OH + Na^+ + OH^-$
3) **Hydrolysis**: When water breaks Si-O-Si bonds to form silanols $\text{-}Si\text{-}O\text{-}Si\equiv + H_2O \rightarrow \equiv Si\text{-}OH + HO\text{-}Si\equiv \rightarrow \equiv Si\text{-}O\text{-}Si\equiv + H2O$.

After the evaporation of the water film, crystalline weathering products can be identified on the surface. They are mainly sulfates, such as gypsum ($CaSO_4.2H_2O$), arcanite (K_2SO_4), and syngenite ($CaSO_4.K_2SO_4.H_2O$), besides some carbonates, nit-rates, chlorides, and organic compounds [15]. In leached or corroded glass, the composition of weathered crusts differs significantly from that of the sample's durable glass core. Alkali content reduces, while silica and calcium content increases [13]. Although it was long assumed that the formation of a significant corrosion layer takes centuries, it has been recently demonstrated that the degradation of certain glass compositions is relatively fast (dec-ades) in the storage environment because of the activity of the pollutants originating from wood cases [16]. The glass weights understudy date back to the Fatimid period (969-1171 AD). They were taken from the excavations of Tell Abu Mina in Alexan-dria in 1995. Since excavation, these glass weights have been stored in uncontrolled condition storage at the Rashid store of antiquities (Rashid is a port city of the Nile Delta located about 65 km to the east of Alexandria), fig. (1). The stored glass weights suffered several aspects of deter-ioration. The main objective of this study is to assess the deterioration state of those glass weights kept at the Rashid store of antiquities according to the storage env-ironment and diagnose the various aspects of decay using visual observation and SEM. It also aims to characterize the chemical composition of the glass weights. It aims to monitor the climatic factors at the Rashid store of antiquities and identify their impact on the glass weights. Moreover, it aims to make a treatment plan to preserve and protect the examined glass weights from deterioration.

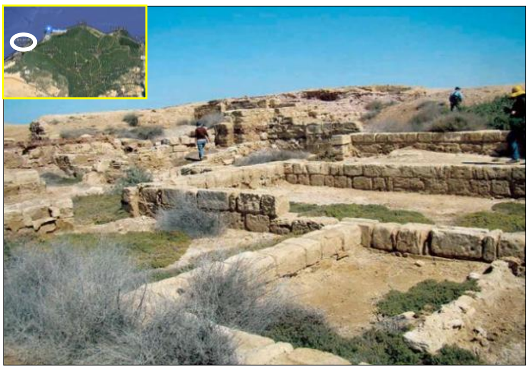


Figure (1) Shows Abu Mina excavation sites.

2. Assessment of the Deterioration of the Glass Weights

It is noted that the surface of the glass weights is affected by the corrosion of glass and many different deterioration processes because they were excavated from damp soil and stored in an inappropriate environmental condition. They were stored in plastic bags and small matchboxes. These materials, which are off-gas volatile organic compounds, can result in damage to glass objects. It is assumed that the glass weights have been stored in this way since their exhumation. No attempt has been made to control their environment. Most glass weights are completely corroded and vulnerable to severe chemical decay. Various damage products were formed resulting from the physio-chemical reactions between the glass and the medium of storage. Glass corrosion causes many different aspects of deterioration that are evident in the case of the studied glass weights, including cracking, dulling, milky–opaque layers, iridescence, and pitting. These aspects are described in this section. * Cracks, fig. (2-a) occur on the surface of the glass because the outer layers of hydrated silica are hygroscopic. These layers absorb and desorb water while expanding and contracting, leading to microscopic surface cracks [9]. * Dulling, fig. (2-b) describes the condition of glass that lost its original clarity and transparency and became translucent. It is easily distinguished from dulling caused by scratches, abrasion, or stains [9]. * Milky–opaque layers, fig. (2-c) opaque white layers on the surface of the glass and maybe interspersed with white lines that are superficial at the beginning. However, in advanced cases of damage, these lines become deeper and extend to the interior part, causing corrosion in the body of the glass [17]. * Iridescence, fig. (2-d) describes a variegated coloration of the surface of the glass. It sometimes occurs alone or with other types of weathering. The thickness of the surface layers containing metal oxides causes light interference, resulting in vivid iridescent colors, such as gold, purple, and pink [18]. * Pitting, fig. (2-e) in advanced cases of damage, the layers of rust peel off and separate. In severe damage cases, they turn into a fine powder, leaving behind small round pits that often contain the products of glass damage known as corrosion pits. Sometimes, those pits are in the form of concentric circles around the starting point and activate damage if the relative humidity rises again [19].

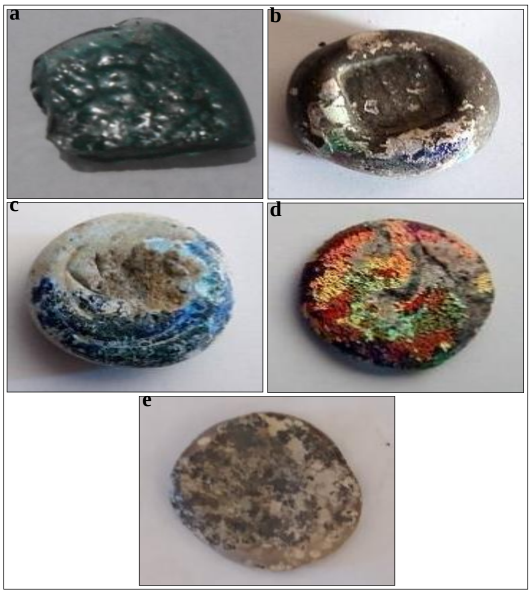


Figure (2) Shows series of deteriorated glass weights stored in Rashid's antiquities store; **a.** non affected glass weights, **b.** weights No.620, **c.** No.575, **d.** No. 143, **e.** No. 1183

3. Materials and Methods

3.1. *Sampling*

The present study was conducted by carefully taking five small samples from weathering crusts, which flaked away from the surface of the glass weights, tab (1).

Table (1) shows the description of the glass weights selected for the analytical study.

Sample Code	Storage No.	Sample Description
A	620	Dark green glass weight, broken edges.
B	575	Brownish glass weight, dull and pitted, with enamel like surface layers.
C	574	Light blue glass weight, with white opaque layer.
D	143	Completely corroded glass weight, with iridescence layers.
E	1183	Light green glass weight, corroded and pitted.

3.2. *Analytical methodology*

The examination and analysis of glass weights reveal much information about their components and state of deterioration. The samples were taken, examined, and analyzed by the following methods.

3.2.1. Examination methods

3.2.1.1. *USB digital microscope*

Leuchtturm USB digital microscope (China) with 20 to 500-X zoom 8 LED lights with measurement software was used to examine the glass surface features.

3.2.1.2. *SEM examination*

Some invaluable corrosion crusts that flaked away and separated from the glass surfaces were collected and investigated by SEM Model Quanta 250 FEG to examine both the compositional phase and the surface structure of the glass material.

3.2.2. Analysis methods

3.2.2.1. *EDX analysis*

An Energy Dispersive X-ray instrument, with accelerating voltage 30 K.V. was used to determine the chemical composition of the corroded glass weights. This method was the most precise for determining the elemental composition and concentration of elements in a sample. It was preferred because it required only a small amount of samples, which suited small glass samples and falling glass crusts.

3.2.3. Storage conditions monitoring

Temperature, humidity, and lighting intensity were measured at the Rashid store of antiquities during summer and winter 2020 using a Hobo Data Logger device to detect their impact on the stored glass weights.

4. Results

4.1. *USB digital microscope*

The examination by USB digital microscope indicated that these glass weights varied in terms of decay rate and nature of corrosion. The surface glass weight, fig. (3-a) was covered with hollows and the outcomes of rust and weathering. However, it was in a fairly good condition compared to the other samples. It likely seems that the weathering crusts found on the surface glass weights, fig_s. (3-b, c) were an extreme form of the stone-like weathering were extremely hard and flinty. The surface of the glass weight, fig. (3-d) was covered by a thick opaque enamel-like weathering surface, which in some areas flaked away to reveal an iridescent layer. The corroded surfaces became very soft, powdery, and easy to destroy, fig. (3-e).

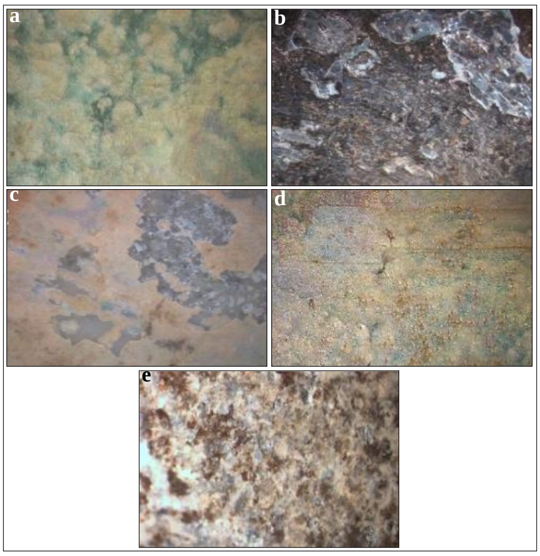
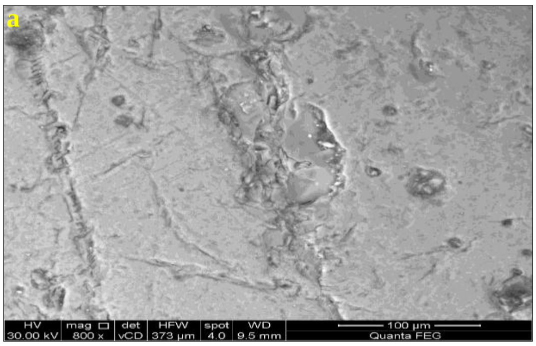


Figure (3) Shows USB digital microscope images of the surface of the glass weights

4.2. *SEM-EDX results*

SEM examination of the glass samples revealed that the surface of the glass weight no. 620 was filled with scratches and cracks, fig. (4-a), and the surface of the glass weight no.575 had a new formation of calcite phase, fig. (4-b). Moreover, the surfaces of the glass weights no. 574, 143, 1184 were corroded entirely, fig. (4-c). Analytical results given in tab. (2) indicate that the major components of the glass samples are silica (SiO_2 avg. 74.54%), soda (Na_2O avg. 5.28%), lime (CaO avg.5.59%), and alumina (Al_2O_3 avg. 4.37%). The samples are characterized by the low content of potash (K_2O avg. 1.94%) and magnesia (MgO avg. 2.29%).



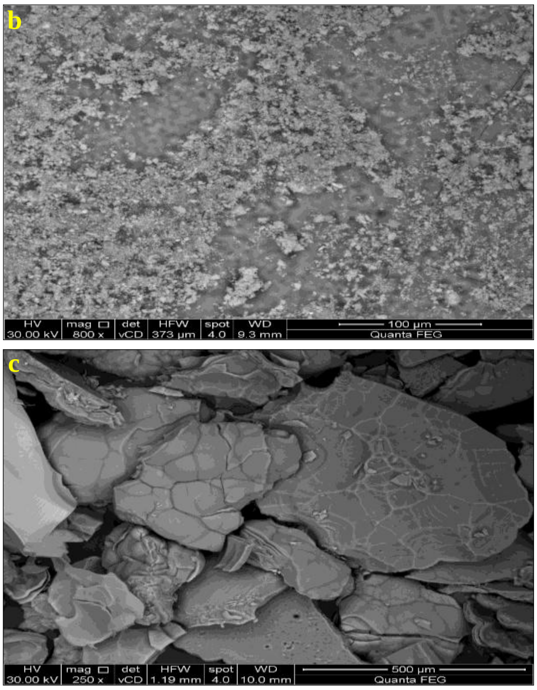


Figure (4) Shows secondary electron images of damaged glass samples; **a.** scratching and cracking glass surface, **b.** formation of calcium carbonate encrustations, **c.** destroyed weathered layers and losses its glassy nature, in addition to large areas of weathering crusts, rich in dissolutions voids, formation of weathering crusts and rich in dissolution voids.

Table (2) The chemical components of the weathering crusts of glass objects by EDX analysis

Sample Code	Oxides									
	SiO_2	Na_2O	MgO	Al_2O_3	K_2O	CaO	Cl_2O	Fe_2O_3	MnO	CuO
A	68.36	8.20	2.88	7.13	2.64	8.45	0.38	1.87	-----	-----
B	72.67	5.16	1.67	4.71	2.43	9.21	1.97	1.17	1.10	-----
C	81.89	3.79	1.45	2.72	0.93	2.62	3.11	2.39	-----	0.83
D	76.74	4.49	2.93	2.45	1.41	2.48	2.26	3.14	2.17	1.93
E	73.04	4.79	2.53	4.87	2.30	5.21	3.43	3.81	-----	-----

4.3. *Monitoring the storage conditions*

The measuring results of relative humidity, temperature, and light intensity during summer and winter 2020 at the Rashid store of antiquities are listed in tab. (3).

Table (3) The results of the storage conditions' monitoring

Variables	Winter season			Summer season		
	RH %	Temp.	Lux	RH %	Temp.	Lux
Max.	85.6	19.3	172	87.5	36.8	230.7
Min.	49.4	16	4.9	50.6	25.8	11.8
Average	67.5	17.5	88.4	69.1	31.3	121.3

5. Discussion

The glass weights suffered from different deterioration phenomena. The results of SEM investigation confirmed that the glass surfaces of these objects seem to be inhomogeneous pitted, curviplanar, and highly fractured. Large areas of weathering crusts were destroyed, rich in dissolution voids, lost their glassy nature with micro-cracks. Other aspects of deterioration were observed, such as the formation of weathering crusts, calcareous salt growing, soiled deposits covering glass surface, sugar-like surface, flaking, and highly fissured nature of decayed crusts. Chemically, these glasses could be classified as soda-lime-silica (Na_2O- CaO- SiO_2); the type of ancient glass that was popular for more than 3000 years [20,21]. This composition reveals that the primary raw materials of the glass were sand, as a source of silica, natron, as a source of alkali soda, [22] and lime, as a source of calcium. The low content of potash and magnesia indicates that the glass samples are natron-based. These glasses are completely decomposed due to the intensive deterioration factors, especially water, for a long time in the ground and the improper storage at the Rashid store of antiquities. There was a high percentage of silica due to the glass component solution and the deposition of insoluble components on glass surfaces. Moreover, there was a low percentage of alkaline soda as a result of the leaching to the surfaces and its solubility during burial in the wet environment. All samples contained chlorine (Cl_2O) because the burial environment consisted of halite (NaCl). Natron was used as a flux for all samples. Iron oxide is present as an impurity associated with sands, almost exclusively responsible for coloring glass to green and yellowish-green [23,24]. The Al_2O_3 and Fe_2O_3 of the corroded surfaces increased obviously due to the dissolution of other glass components, leaving an apparent enrichment of Al and Fe, which have very low solubility. The percentage of manganese oxide was observed in samples B and D (MnO 1.10% and 2.17%, respectively), indicating that manganese was intentionally used as a colorizing agent in the form of (Mn^{+3}) ion to color the glass purple or violet [25]. In all other transparent and colorless samples, manganese was utilized as a decolorizing agent in the type of (Mn^{+2}) ion, performing as an oxidizing agent, transforming the iron from its reduced state (Fe^{+2}). It has a strong greenish blue colorant to its oxidized state (Fe^{+3}) with a yellowish but much less intense colorant [23]. Monitoring the storage conditions showed that the average relative humidity was 69% in summer and 67% in winter. It is assumed that the relative humidity of the stored glass objects does not exceed 40% [26,27]. Therefore, those high percentages negatively affected the glass objects, especially the unstable glass. Sodium and potassium are slightly soluble in some glass compositions. In the presence of high relative humidity, these components can reach out to the surface of the glass, where they are converted to carbonates. These carbonates attract moisture, and tiny droplets of water begin to appear on the surface of the glass [28]; hence, it is known as weeping glass. Further leaching and droplet formation stop if the glass is kept at a relative humidity below 40% [26]. It is observed that the average temperature was 31 °C in summer and 17 °C in winter. The recommended temperature level is 18-20 °C (64-68° F). The temperature should not exceed 24 °C (75° F) because fluctuating temperatures could cause (crizzling glass), which were small cracks on the surface of the glass and weakened its strength and durability. After all, they expanded and contracted rapidly, setting up destructive stresses in the object [29,30]. The average light intensity was 121.25 lux in summer and 88.4 lux in winter. The energy in light reacts with the molecules in glass objects causing physical and chemical changes. It is assumed that light in storage areas should be kept at a minimum (complete darkness). The sources of natural light should be eliminated when possible or filtered when elimination is not possible. Windows should be covered with shades, drapes, or blinds [31].

6. Treatment Plan of the Deterioration Aspects

Dirty layers and soil deposits should be removed carefully by mechanical cleaning. As water and moisture are the essential factors of initiating and sustaining forms of glass decomposition, even the damage caused by dehydration is a consequence of a previous attack by water. Therefore, wet cleaning using water is excluded, and chemical cleaning using organic solvents should be applied. Cotton swabs saturated with acetone can be topically used for delicate cleaning work. It is necessary to use the neutral solution of the disodium salt of EDTA (PH7) at 5% concentration by brushing technique [26]. This treatment aims to stop the continuity of corrosion reactions and prevent any expected devitrification [32]. After that, it is possible to strengthen and isolate the fragile archaeological glass using 1% Nano zinc oxide dispersed in 3% Nano paraloid [33]. This nanocomposite material is considered the best material for the consolidation and protection of archaeological glass because it is a super hydrophobic and self-cleaning protective material [34,35]. Then, these glass weights should be preserved in stable and appropriate conditions. Suitable storage materials that do not emit gases and acids that can cause damage to glass objects should be used. It is recommended to store the glass weights in small resalable bags of polyethylene for each and keep them vertically inside cardboard containers or acid-free boxes provided with silica gel to absorb the moisture and prevent the glass weights from further corrosion because the storeroom is very damp. Glass weights should be stored in a stable environment as possible. Ideally, the relative humidity should be 40% or less [26,27].

7. Conclusion

Burying glass weights in Abo-Mina, Alexandria and keeping them at the Rashid store of antiquities harmed them because these areas are coastal and contain high humidity. This condition was evident in the examination of the naked eye and SEM. The glass weights were completely corroded and subject to intensive corrosion and other deterioration aspects, such as pitting, cracking, encrustation, iridescence, dulling, and salt crystallization, which led to damage and loss of the decorations and inscriptions on the surface. The chemical analyses indicated that the analyzed samples are examples of soda-lime-silica glass, with natron used as a flux, which was mostly obtained from Wadi El Natrun in Egypt. Al_2O_3 *and* Fe_2O_3 *of the corroded surfaces also increased,* *obviously due to the dissolution of other glass components, leaving an apparent enrichment of Al and Fe, which had very low solubility. Also, the percentage of iron ions may be added to obtain a green color to the glass weights. Also, a high percentage of silica was observed due to the deposition of insoluble components on the surfaces of glass during the process of leaching alkaline substances in the presence of high humidity and decreased alkaline oxides content. Monitoring RH, T, and light at Rashid store of antiquities during sum-mer and winter showed higher proportions than the permissible ratios for preserving the glass antiquities. Thus, glass weights should be packed in an airtight container with silica gel and stored in suitable boxes or cardboard containers. It should be taken into account that the humidity in the storage place does not exceed 40%.*

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