Nel blu, dipinto di blu,
Five Centuries of Blue Roman Glass from the Palatine Hill:
A Multi-Analytical Approach to Provenance and Production Technology

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A mi madre y mi hermana, por siempre estar.
A Fer e Irina, por recordarme que siempre se puede encontrar ángeles.
A Ed, Stef, Chris, Marta, Javier, Marilena y Amanda que me ayudaron a ver
nel blu, dipinto di blue *durante la cuarentena.*

Abstract

Based on a multi-analytical approach, this research aimed to characterize and compare the glass-making processes and provenance of the Roman blue glass collection, dated from the the 1st century to Late Antiquity, found in the Helagabalus baths in the North East Palatine archaeological site. The samples were analysed by Optical Microscopy and SEM-EDS to identify homogeneity, glass morphological features like bubbles, inclusions and corrosion. EMPA was applied to determine the elemental composition of major and minor elements, whereas LA-ICP-MS allowed us to define trace elements. Major and minor elements analysis, performed by EMPA, gave particular information regarding the ingredients (network former, flux, colourants and decolourants) used in the glass batch. SEM-EDS and LA-ICP-MS in combination with EMPA allowed us to group the collection for provenance and technology information. Comparison of the data with the literature allowed to identify the raw materials provenance. This study highlighted changes in glass-making technology and provenance from the 1st to 5th of the Roman blue glass collection. The particular study of this collection can give further information about the glass making techniques of that time.

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Chapter 1

Introduction

Glass artefacts and waste are commonly found in Roman archaeological contexts. It has been object of archaeometric studies, proven to be effective in giving valuable information regarding trading routes, workshops techniques and firing technology of the respective time period[1,2]. Although Roman glass has been found all over the sites belonging to the Roman period, it is believed that the sources of the raw materials and colorants were limited[1,3–5]. Further archaeometric studies continue to contribute to the construction of the big picture regarding provenance, trade and technology of glass of the same time span.

The importance of cobalt-blue glass, characterization and provenance studies resides in several reasons. Roman blue glass is a prevalent colour (with green) through the 2nd to the 4th centuries in archaeological records[6]. It presents a similar composition through this period of time but the information regarding cobalt sources supplied is not clear[7,8]. Due to evidence of blue-coloured glass chunks from the 1st century found around the Mediterranean[9], blue glass could have been coloured at a primary location[9] as in earlier times[10]. These could make a direct link between compositional families and the colorants sources.

In order to achieve comparable results, a combination of multiple analytical techniques has been proven to be effective for glass characterization[11–15]. In general, glass is chemically analysed to identify the possible function of each element and relate it to its manufacture and the raw materials. Some of the most common techniques for glass elementary analysis are X-ray Fluorescence (XRF)[16–21], Scanning Electron Microscope with Energy Dispersive Spectroscopy (SEM-EDS)[1,22–28], Electron Microprobe Analysis coupled with Wavelength Dispersive Spectroscopy (EMPA-WDS)[29,30,30–32] and Inductively Coupled Plasma Mass Spectrometry (ICP-MS)[33–35].

The spatial resolution of SEM-EDS has been used to chemically character-

ize bulk material, morphological features and micro-texture of corrosion layers in glass samples, to obtain information on production techniques and decay mechanisms[22–24,26,27]. With opaque glasses, SEM-EDS qualitative analysis has been used to reveal the presence of encrusted crystals (for example SnO_2) dispersed in a lead-reach matrix of samples[31,36,37]. The application of EMPA on glass to study the chemistry of bulk material is frequent, some examples are: coloured (white, green, pink, blue, yellow, red) stained glasses[38], 16th century glasses[39] and Roman glasses[29,32,40]. Major and minor elements analyses, performed by EMPA-WDS, can give particular information regarding the ingredients (flux, opacifiers or colourants) used in the glass batch and lead to typologies of raw materials[30,31]. Both techniques are used for imaging, qualitative and quantitative analysis. SEM-EDS is optimized for imaging, especially when high resolution images are needed, whereas EMPA is designed primarily for quantitative analysis.

Several techniques and methods have been applied in provenance studies including trace elements analysis[7,8,33], including rare earth element patterns[22,23,33,41,42], and isotopic signatures (e.g., $^{87}Sr/^{86}Sr$[43,44] and $^{143}Nd/^{144}Nd$[44]) as they may be geological markers of the raw materials[4,4,45,46]. Due to its low detection limits ICP-MS has been widely applied in trace elements studies[4,7,35,42,47] and when combined with Laser Ablation, ICP-MS can become an alternative that involves direct solid sampling with minimal invasion[35]. Proper correlations between trace elements in glass have allowed to identify raw material provenance[7,48].

A collection of deep blue glasses was selected from the assemblage of the Palatine North East (PNE) site, inside the Archaeological park of the Roman Forum-Palatine Hill, Rome. The collection has samples dated from the late 1st BC to the 5th AD century, belonging to the Helegabalus Baths context. Previous studies of glass collection from the PNE archaeological site regarding colorless samples[27] and glass waste[29] have provided important contributions to cultural and chronological framing of this archaeological context.

Moreover, the importance of the site within the Roman world, and its continuous occupation (from the early Iron Age to the Middle Ages) gives this blue glass collection a high variability of time spans and probably provenance, making it suitable for a possible comparison of glass making technology through time. An interdisciplinary combination of the multi-analytical characterization and the available archaeological information could clarify and expand the already existing awareness of Roman trades and supplies.

Based on a multi-analytical approach, and relying on the current knowledge of Roman blue glass, this research aims to do a provenance classification and a time lapse comparison (from the 1st to 5th century) of the glass-making processes of the Roman blue glass collection, found in the Helagabalus baths in the NE Palatine archaeological site. A combination of imaging, spectroscopic and spectrometric analyses will be applied. Samples will be analysed by Optical Microscopy and SEM-EDS to identify homogeneity, glass morphological features like bubbles, inclusions and corrosion. EMPA will be use to determine the elemental composition of major and

minor elements, whereas LA-ICP-MS will allow to define trace elements. Major and minor elements will give particular information regarding the ingredients (network former, flux, colourants and decolourants) used in the glass batch. SEM-EDS and LA-ICP-MS in combination with EMPA allow to identify raw materials provenance and technology information.

Overview

First, in Chapter 1 intends to give a summary that includes basic concepts of the vitreous state and its main properties. There are two main topics to be outlined, What is Glass?, and second, the conditions that a substance must meet in order to be in vitreous state. Some characteristics of the chemistry of glass making will be addressed, for example, the process of glass melting and function of each constituent of the batch (flux, stabilizer, opacifier and colorants) will be described.

In the second place, in Chapter 2 an overview of Roman glass history and technology taking on account historical resources (including Pliny and other Roman historians), archaeological evidence, and archaeometric analysis will be presented. Starting from a short review of the history of glass from what is believed to be the beginning of the use of glass, until the fall of the Roman Empire. Taking on account the centralized glass making system, a description of primary and secondary workshops, as well as the possible main sources of the raw materials, including sand, soda and the colorants, will be included.

A more detailed description of the history and archaeological chronology of the PNE archaeological site will be given in Chapter 3. This section will intend to highlight the importance of the site and the and high variability of possible dating and provenance that the blue samples could have. The second part of the thesis will describe the multi-analytical methodology applied to the collection, including SEM-EDS high resolution imaging and qualitative analysis, and the EMPA and LA-ICP-MS elemental quantification. The analyses and comparison of the data with the literature will allow to identify the raw materials and its provenance.

Chapter 2

Vitreous State

2.1 Definition of Vitreous State

In a solid, the atomic structure is thermodynamically stable and the chemical forces are strong enough to keep that structure cohesive even when it is not confined in a receptacle[49]. Solids can be crystalline or non-crystalline. A crystal has a well-ordered atomic structure on a periodic lattice in short and long range, unlike its non-crystal counterpart. Non-crystalline solids can be divided into two thermodynamically distinct classes: glasses and amorphous solids[50]. 'Glass' satisfies the condition of short range order (or 'coordination polyhedra') even when it lacks of long range periodic atomic arrangement. Glass can be formed in a melt-cooling process were the structure of the melt is frozen-in, without crystallization. This transition is time-dependent and occurs over a temperature range known as the glass transformation region[15].

If a liquid can be cooled below the melting temperature of the crystal without crystallization, a supercooled liquid (SCL) is obtained[15]. A SCL is metastable, it can relax and crystallize at any positive temperature with sufficient time, which strongly depends on its chemical composition and temperature[51]. The structure of the liquid continues to rearrange as the temperature decreases, but there is no abrupt decrease in volume due to the discontinuity of the new structure. If temperature changes at a sufficiently high rate, the viscosity will increase until the molecules will no longer completely rearrange to the equilibrium liquid state; instead, the structure will become fixed, with no order. According to Zanotto and Mauro[51] glass is a "*...non-equilibrium, non-crystalline condensed state of matter that exhibits a glass transition. The structure of glass is similar to that of its parent supercooled liquid (SCL), and it spontaneously relaxes toward the SCL state...*". In the limit of infinite time, its fate is to crystallize but the energy required to jump from a meta-stable to a stable state (crystallization) at room temperatures could hardly occur[52].

It was calculated that the time needed for that the change of shape of a typical

window glass (K_2O–Na_2O–CaO–MgO–Al_2O_3– SiO_2 plus impurities) be appreciable for the human eye. The relaxation time, at room temperature will require over geological periods, not within the limits of human history (around 10^{32} years and well beyond the age of the universe 10^{10} years).

2.2 Criteria for Glass Formation

There are conditions that a substance should meet in order to transform into vitreous state. The main criteria for glass formation can be based on intrinsic (geometrical, structural, chemical bonds and thermodynamics of the system) or extrinsic features (kinetic cooling rate)[1]. Although it is well known that it is possible to make glass from almost any material, in this section the developments of glass formation criteria will be explained in order to better understand the behaviour of the most common glass: silica glass.

In 1932, Zachariasen[53], extended the ideas of Goldschmidt and made some statements on the formation of oxide glasses:

1. Each oxygen atom must be bonded with no more than two R cations. Higher coordination numbers for the oxygen cations prevent the variations in cation-oxygen bond angles necessary to form a non-periodic network.
2. The number of oxygen atoms surrounding the central cation must be small, specifically either 3 or 4.
3. The coordination polyhedra formed by either oxygen triangles or oxygen tetrahedra must share vertices, not edges or faces.
4. The network can only be 3-dimensional if at least three vertices of each coordination polyhedron are shared with other neighbouring polyhedra.

(a) Crystal

(b) Glass

Figure 2.1. Comparison between the dimensional structures of a glass and a crystal of the same compound R_2O_3. Scheme from Zachariasen[53].

These conditions make possible the formation of a continuous, disordered, open structure that favours network deformability without too much stress on bonds. In figure 2.1 it is shown a two dimensional scheme of randomly distributed units of a glass, forming a distorted network and the geometrically and periodically ordered regular network of the same compound in crystalline state.

The process of obtaining glass depends on different factors and some are closely
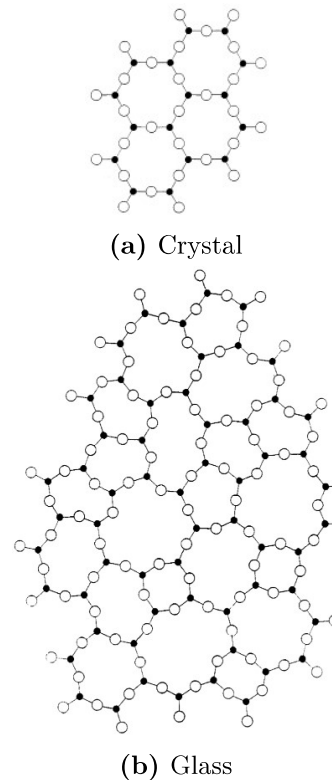
related. The understanding of glass formation through different criteria could help to comprehend some typical characteristics of glass behaviour to better study it. Some examples of formation criteria are:

- The cations of the molecules need to have a low coordination number[53,54] and high network connectivity to enhance the structure.
- Cations with the highest field intensity are the best glass forming atoms, while those with low field intensity behave as glass modifying atoms (see section 2.3.2)[55].
- The best oxides to form a glass are those with a high single bond strength between the cation (R) and the oxygen, as well as a low melting temperature[56].
- Mixed types of bond co-existing favours the structural disorder, substances with a mixed covalent chemical bonds (53–68% covalent) are suitable for glass-forming.[57]
- Thermodynamical criteria[58].

2.3   Silica Glass

There is a wide variety of substances of different chemical nature that can be in vitreous state. Components and proportions can vary since glasses do not follow stoichiometric ratios. Nevertheless, glass families can be defined according to the main component of each glass network. The most common and probably larger group is the one of oxide glasses. And, as foreseen, silicon oxide (silica) meets all the requirements listed above. Amongst all oxide glasses it is the most important and most common in ancient glasses, especially soda-lime silicate glasses.

Silica is formed by SiO_2, and usually arranges itself in a continuous network of $[SiO_4]$ tetrahedra with a silicon in the middle and 4 oxygen ions (see fig. 2.2), its vertices acting as bonding bridges between other tetrahedra to ensure network continuity.
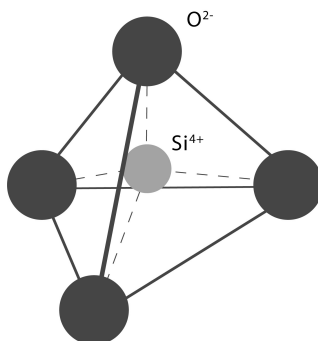

Figure 2.2.   Model of a $[SiO_4]^{-4}$ tetrahedron, with an O-Si-O bond angle of 109. 5°[59].

For a pure silica glass, the coherent structure without network discontinuity points and the strength of the Si–O bonds determines the low thermal expansion

coefficient, high viscosity and high optical transmission range in the UV region. However, due to the high melting temperature at around 1723 °C (even at $\equiv$ 2000°C the melt is rigid and difficult to shape) it is known that it is necessary to add other compounds to change the properties of the melt so that it can be worked[15].

Regardless of the source of the components used to produce a specific glass, according to Shelby[15] the batch materials can be divided into five categories based on their function: glass-former, flux, property modifier (or stabilizer), fining agent, and colourants (including opacifiers). It is possible that the same compound belongs to different categories. For example, alumina can serve as a glass-former , but it is also considered a property modifier in most silicate glasses[15]. In the next sections these categories will be discussed only for silica glasses where the glass or network former is made of SiO_2 tetrahedra.

2.3.1   Fluxes

To reduce the melting processing temperature within practical limits ($<$ 1600 °C), fluxes are added to the batch. The most common fluxes are alkali oxides, especially Na_2O (soda) and K_2O (potash), but also PbO[15]. The incorporation of these oxides into the structure induces partial network opening. Each alkali oxide molecule added to the glass network forms two non-bridging oxygens neutralising its own positive charge (see figure 2.3). Sodium ions located in the interstitial sites of the network surrounded by oxygen ions, inducing an increasing breakdown of siloxane bonds $\equiv Si - O - Si \equiv$ and, therefore, a progressive weakness of the glass structure. This latter implies a decrease of the melting point and the viscosity of the batch and at the same time an increase in both, its thermal expansion coefficient and crystallization tendency. While addition of fluxes to silica leads to some convenient properties, the addition of large amounts of alkali oxides results in serious degradation in many others, like chemical durability[15].

2.3.2   Stabilizers

The degradation process produced by alkali oxides can be countered with the addition of stabilizers which include aluminium, alkaline-earth, and transition metal oxides. The incorporation of alkaline-earth oxides, for example CaO, into the glass network takes place in a similar way to alkaline ones. In this case, each Ca^{2+}-ion will bond to two non-bridging oxygens counteracting the breakdown of $\equiv$ Si-O-Si $\equiv$ bonds[1].
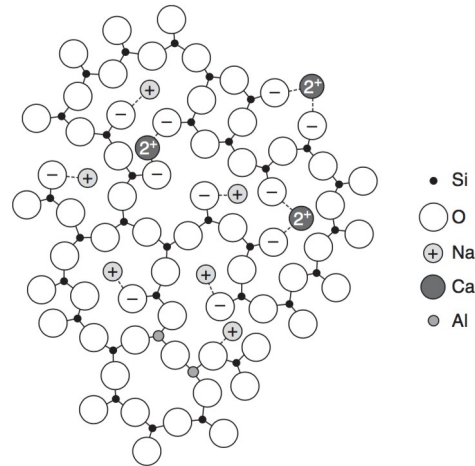
Figure 2.3.   Planar plot from the structure of a soda-lime glass with a low content of Al2O3. Source: Janssens[1]

In conventional glasses SiO_2, Na_2O and CaO are the major components. In addition other components such as K_2O, MgO and Al_2O_3 are usually present in the raw materials as impurities. A glass with enough amount of stabilizer can avoid devitrification.

Another example is Al^{3+}, when its ions are present in low proportion, they can coordinate, as $[AlO_4]$ (alumina) tetrahedra, with the $[SiO_4]$ groups in the glass structure and replace them in the role of network formers. Since the oxygen/cation ratio in Al_2O_3 (3:2) is smaller than in SiO_2 (2:1), when Al^{3+}-ions are surrounded by four oxygens they have to incorporate one non-bridging oxygen to complete their coordination, these non-bridging oxygens become "bridging oxygens" and the structure connectivity increases. The negative charge excess in the $[AlO_4]$ groups is compensated by the positive charge of the alkaline ions that are located close by (see figure 2.3), and as connectivity increases, also stability does. For this to happen, the molar concentration of Al_2O_3 must be lower than the alkaline oxides (X) $([Al_2O_3]/[X_2O]) < 1$, so that the number of non-bridging oxygens of Al^{3+}-ions is low enough to accomplish tetrahedral coordination. On the contrary Al^{3+}-ions will play the role of network modifiers and their coordination will be octahedral[1].

When a compound ceases to be a network modifier to become a network former, it is no longer considered a silicate glass and its physical and chemical properties change. Some examples of binary glasses are alkaline silicate (concentration of alkaline elements > 10% mol) or lead glass. Although Pb can function as a network modifier and as a network former according to Wang and Zhang[60] for concentrations less than 40 mol% it behaves like a network modifier. Hence, stabilizers not only partially counteract the reduction of the melting and glass transition temperatures (obtained by addition of fluxes), but they can change other properties[15,59].

2.3.3 Fining Agents

Finally, when melting the batch in modern day workshops, there are certain additives that are known to promote the removal of bubbles from the melt. Fining agents include arsenic and antimony oxides, potassium and sodium nitrates, NaCl, fluorides such as CaF_2, NaF, and Na_3AlF_6, and a number of sulfates[15]. These materials are usually present in very small quantities (< 1 wt%), and they have minor effects on the optical properties of the final glass, for example the addition of these additives, on purpose or as an impurity, can produce a final product without unwanted trapped bubbles[15].

2.3.4 Colourants

The phenomena that gives colour to glass can be, the reflection of the light at the glass–air interface and the absorption or scattering of light in the bulk glass. Colourants are elements present in small quantities responsible of these phenomena. Particularly, in ancient and historic glasses, the three ways found to produce the colour effect described by[61] are: metallic oxides, colloids and opacifiers.

Presence of transition metal ions in the batch can result in colour of different hues and intensities. The colour depends in position and number of absorption bands characteristic of each ion, its electronic configuration[15,25]. The 3d electronic levels are identical in energy to free ions, however when they are surrounded by few anions in the glass, the interaction of the electric fields causes a small splitting of the energy levels. The energy differences of the cation electron fields are commonly in the range of 1 to 3 eV[15] resulting in visible colouration. In table 2.1 some examples are shown. That process is similar for the 4f electronic levels of the rare earth ions.

The addition of colloidal suspensions of certain metallic (such as silver for yellow, copper for orange or gold for red), or other insoluble particles in the batch would cause colouration due to the intense optical absorption by these particles. The same way inclusion of opacifying agents which produce opal and translucent effects.

Table 2.1. Transition elements in soda-lime–silica glasses and the colour they produce from Janssens[1]

Ion	Coordination	Wavelength (nm)	Colour	Period	
Cr^{3+}	Octahedral	450, 650	Green	19th Century onwards	
Mn^{3+}	Octahedral	490	Red, purple	Ancient periods up to today	
Fe^{2+}	Octahedral	1050	Blue	Ancient periods up to today	Janssens[1]
Fe^{3+}	Tetrahedral	435, 420, 380	Yellow		
Co^{2+}	Tetrahedral	530, 590, 645	Blue	Ancient periods up to today	
Ni^{2+}	Octahedral	450, 930, 1800	Brown	19th Century onwards	Janssens[1]
	Tetrahedral	560, 630, 1200			
Cu^{2+}	Octahedral	780	Green/Turquoise	Ancient periods up to today	

The production of coloured glasses not only depends on the metallic oxides present

in the batch, but also on the temperature and state of oxidation or reduction in the furnace. In any case, the exact composition of ancient glasses was complex, the exact quantities of raw materials and furnace conditions were unknown, so it is probable that the final results were not accurately determined[61]. Additionally opacifying the glass was a common practice used to change the glass hue. Opacification could occur by the presence of crystals (formed *in situ* od externaly added) or by a high number of bubbles[62].

Chapter 3

Roman Glass

3.1   Review of the pre-Roman History of Glass

It is well supported by archaeological evidence that the first man-made objects of glass were made from obsidian. During the Paleolithic, the black, natural, volcanic, glassy material was used to obtain sharp tools and resistant objects[61,63–65].Based on archaeometric studies, it has been possible to designate provenance to the material of these items and even determine long distance trade routes, e.g. around the Mediterranean basin[66].

Later in time (back to the middle of the 3$^{\text{rd}}$ millennium BC), a vitreous paste in the form of faïence found in Mesopotamia, seems to precede the use of glazing in upper Egypt[67], before glass as we know it, appeared. The ancient Roman historian Pliny the elder suggested that Phoenician merchants had made glass for the first time by accident in the region of Syria[68], although it is more likely that the first glasses were simply slags from copper metallurgy[69]. Some raw glass chunks, dated to the late 3$^{\text{rd}}$ millennium[70] were found in Iraq and Syria at the sites of Eridu, Eshnunna, and Tell Brak[71], southern Mesopotamia, and according to the archaeological evidence, it is possible that glass could had been produced deliberately in that moment.

During the 15$^{\text{th}}$century BC, during Tuthmosis III reign, glass-making centres were created in Egypt, where they flourished until the 20$^{\text{th}}$century B.C.[1,69]. Typically the glass containers (mainly cosmetics and ritual vases) were obtained by forming a mixture of clay and horse dung applied to the end of a metal rod, then, molten glass was gathered around the core, and after cooling, the core was removed by scraping (core-forming technique). After the decline of Egyptian production, between 1200 and 900 BC, there are few archaeological sites with vitreous discoveries[67,69].

In the 9$^{\text{th}}$century BC, evidence shows higher production of glass objects, especially in the Mesopotamia and Syria-Palestine areas. Findings of glass material in the archaeological sites of Anatolia, Babylon and Assyria (particularly in Nin-

eveh)[72,73] were accompanied by the discovery of tablets with information regarding the preparation of various materials, including glass. The oldest technical description of glass production was found in the library of the Assyrian King Ashurbanipal. A group of clay tablets details the mystical rituals surrounding the production, and the recipe for glassmaking. The four basic ingredients were immanakku-stone (sand), naga-plant ashes (soda ash), a 'white plant' (unknown ingredient) and a 'slow copper' compound (maybe used as a colourant)[72,73].

Throughout the 8th-7th centuries BC some working techniques were developed (*lost-wax*-produced cups and *mosaic* were found). During the second half of the 8th century, the molten vases on a friable core reappeared again in Mesopotamia. From the 7th century BC, the *core-formed* vases extended from western Asia to the eastern and central Mediterranean regions as a consequence of the settlement of craftsmen from the Near East in Rhodes, Etruria and Istria, near Italy[74,75].

Despite its wide diffusion around the empire it is believed that even by the 5th and 4th century BC, glass was still considered a status symbol[75].After Alexander the Great of Macedonia conquered the lands of Mesopotamia and farther East, the Syrian-Palestinian coastal area, particularly Alexandria became one of the principal production centres of Hellenistic glass.[76] Up to the 1st century BC, the production of glass objects remained substantially limited to this area until around half of the first century BC. Then, around the half of the century a great technological innovation occurred: glassblowing.

The first trade of blown glass is contemporary with the conquest of Egypt (31 BC) and the establishment of the principality of Augustus at the end of the 1st century[77]. Political and economic factors, the intensification of maritime trade in the Mediterranean with the addition of the technical novelty, caused the wide scale spread of glass objects. In the Western world, the Roman Empire helped in establishing numerous glass centers, Syrian and Alexandrian glass blowers were working in Rome and some provinces and the technique expanded until it reached Spain, the Netherlands, Gaul and Brittany[69]. Prices reduced considerably and the use of blown glass containers became popular, up to their predominance on the markets during the 1st century AD.

3.2 From the Augustan era to the Fall of the Western Roman Empire

During the Augustus Age a new world market[78] with all types of commercial activities was created. There are many references in historical documents that show that glass was exported to many different areas of the Empire, often in large quantities[48,79]. Aside historian like Pliny[68] and Strabo[80] further literary evidence of glass production comes from the Edictum de Pretiis Rerum Venalium (Edict on Maximum Prices) issued by Diocletian in 301 AD and in the Historia Augusta (Augustan History). According to Degryse there is a short mention of Alexandrian

and Judaean glasses by name in the first one, and the Egyptian production in the latter[48]. It is possible to conclude from both that the glass was worked in Alexandria but that the manufacture from raw material did not happen there[48,81].

As for the archaeology and archeometric analyses of different contexts, it was shown that there were a limited number of different types of glass compositions circulating the Mediterranean and Northern Europe during the second half of the 1st millennium AD[9,82]. Two main groups were identified, high iron and manganese (HIMT) and Levantine I with lower levels of iron, magnesium and sodium. This last group is similar to earlier blue-green Roman glasses, except for less sodium concentration[70]. The conclusion was that low-iron Roman glasses used sands from the Syro-Palestine region (probably Mouth of Belus River) and later high-iron glasses were probably manufactured using sands from Egypt (possibly Nile Delta surroundings)[83]. Some clear patterns in the composition of Roman glass are well studied, for example, glass from the 4th century (and later) is higher in iron, titanium and manganese than Roman glass of the 1st to 3rd centuries[6,9,84–86]. In this case the high-iron 4th-century glasses were geologically similar with the ones of Egyptian origin[85].

For the 2nd and 3rd centuries composition (with low levels of iron, phosphorus and titanium) the difference in the composition was not only attributed to the presence of antimony and/or manganese (probably added deliberately as decolourisers), but the trace elements also suggested a use of a different sand, therefore a different centre of production. This was also true for blue-green glass (typical from the late 1st to the 3rd centuries). It tend to contain higher levels of iron and other trace elements (like copper) than does colourless glass, suggesting the use of less pure sand or recycling. Isotopic analysis suggests that there may have been a few different production centres for these blue-green glasses and some may not have been in the Eastern Mediterranean. Even studies on colourless glasses (purer sand, less recycled) showed subtle but different compositional groups, not based on the decolourises alone, explained by a certain organisation of glass production within the Roman world[70].

Material analyses and a series of important archaeological discoveries in recent years have built a solid theory of hierarchical production systems during the Roman period. Compositional consistency of different groups can indicate a common production location for each "colour"[9] and could point out at a model of centralised glass production[70], where, large tank furnaces called the primary workshops, mainly concentrated on the Syro-Palestinian coast, would prepare raw material. Then, the product was then sent in blocks to all secondary workshops, where they will re-fuse the material[87].

3.3   Production System

3.3.1   Primary Workshops

A primary workshop was a site outfitted with structures designed for the fusion of raw materials for the production of primary glass which would then be broken into chunks and transported to other workshops across the empire. Admittedly, the archaeological evidence of primary production centers in the Roman period is limited[88]. Some of the few related discoveries are dated between the 4th–8th centuries, and showed that large quantities of natron glass were still made in Egypt and Syro-Palestine area ([9,74,86,88,89]. Some examples in Israel are the remains of 17 rectangular furnaces dating from 8th century AD in excavations at Beit Eli'ezer,[89–91]; four similar furnaces assigned to an earlier period (6th-7th AD) in Apollonia[90,91] and some evidence of primary and secondary production (but no furnace) was found at the site of Horbat Biz'a[92].

For the first three centuries, there are remnants or furnaces discovered on the shores of Lake Maryut, near Alexandria, Egypt (from the Imperial period to the 8th century AD)[93] or near the sand sources, such as the mouth of the Belus River, or close to the alkali sources, such as the Wadi Natrun, Egypt[89,93]. According to Degryse et al.[88] this agrees with the historical sources but little evidence of primary glass factories has been found in the other regions mentioned, for the earlier Roman period. There are some examples that suggest evidence of primary production outside the Syria-Palestine area, like six glass factories from the late Imperial period (c. 4th century) at Hambach[46,94], as well as a 2nd century AD tank furnace in a Roman military camp at Bonn[41], both in Germany.

As for the workshop operation, although the furnaces related to Roman period are different in style from the later found in Israel, it is believed that the procedure of the facilities would be similar. The 17 tanks found in Israel were large and it is estimated that they could produce 8-9 tons of raw glass per firing[89,91]. It is considered that each structure would only have been used once. For a two-weeks process, the raw material would be fused and at the end the kilns were dismantled to extract the large blocks of glass. Subsequently, new furnaces were built nearby. In general, primary workshops were built close to the raw material sources, and would work until the available fuel in the area was exhausted[87].

3.3.2   Secondary Workshops

According to Saguí[87], secondary workshops were small "[...]modest kilns, designed to carry out the simple re-fusion of raw glass with metallic salts, or oxides, for colouring, bleaching, or inducing opacity. These workshops would have also been used to recycle glass...". Meaning that, due to the evidence of secondary glass production (fragments of ingots, crucibles, small circular structures) found close to urban settlements, experts believe that ancient glass production was carried out on

small-scale, compared to the primary workshops.

3.3.3   Recipe and Raw Materials

Despite the fact that glass is a complex material which changes its mineralogic composition during melting, it is possible to assign the origin of the raw materials from the bulk and sometimes even the primary glass center of production[48]. It has been demonstrated the presence of a limited number of glass groups corresponding to primary production centers in the early Roman period[9,86] as well as late Roman (5th–7th century) from the eastern Mediterranean[86,89,95]. There are basically two methods for provenance evaluation. First, comparing the chemical composition of the unknown samples with the composition of known material,or studying the chemical and isotopic data from various Roman glass assemblages to determine the likely locations of glass-making (therefore, probable primary furnaces)[33,48,88]. The isotopic ratios of Sr and Nd have proven to be effective for this matter[4].

It is known that, during Roman period the vitrifier used in glass was silica coming from sand which also provided calcium oxide to stabilize the added flux in the form of soda (rarely potash)[33,48,87]. This mixture is typically called soda-lime glass. The sodium oxide component used, came from sodium carbonates (soda) in a mineral known in Arabic as, 'natron' and was the only one used until the 8th/9th[87]. Lime (calcium carbonate), although naturally found in the form of lime-stone and marine shells, it is suggested that is was not added on purpose, instead, an adequate type of sand with certain proportions of calcium carbonates would be suitable for glass-making. The proportion in the composition of sand some minor impurities, such as aluminium, magnesium and iron oxides, can change the properties of the final product, for example, less than 1% of iron gives a distinctive blue colour[96].

3.3.3.1   Soda

Natron is a naturally occurring deposit of mostly sodium carbonate decahydrate ($Na_2CO_3 \cdot 10H_2O$), sodium bicarbonate ($NaHCO_3$) and small quantities chlorides and/or sulphates[97]. It was the most used flux for vitreous production from the early 4th millennium BC untill 8th-9th AD[87,97]. The Roman historian Pliny the Elder made reference to the substance that would be added to the batch in order to make glass. Particularly, he pointed out that the soda-deposits used to exploit natron were found in Egypt, found in the regions around Naucratis and Memphis.[68,96]. The most known and studied natron deposits in that area are a group of small lakes found in the Wadi el Natrun depression, around 100 km northwest of Cairo and at the lakes of Al-Barnuj at the Nile Western Delta[87,90,96,97]. The mineral would crystallize through evaporation during winter months[98] (Shortland 2004) and would be gathered from the edge of the lakes in the Spring, possibly by breaking lumps from the crust (see 3.1).

During the glass-making process, excess sodium chloride and sulphate have a

limited reaction with silica and form an immiscible layer that would then be skimmed off[97]. However, this last is reflected in the composition where typically Roman glass contains 0.5-1.2% Cl and 0.2-0.5% SO_4 [33,99].

Figure 3.1. Natron deposits on the edge of a soda lake at al-Barnuj, Nile Western Delta, Spring 2004 (photo by I. C. Freestone[96])

3.3.3.2 Sand

As in the case of natron, the source of sand for glassmaking was mentioned by the historians Strabo[80] and Pliny[68] suggesting Syro-Palestine and Ptolemais sands. Another historian, Tacitus coincides with Pliny mentioning a beach near the mouth of the Belus River which meets the sea near Ptolemais as a source of glass making sand[68,100]. Although research is still going on, enough analyses conclude that the sand from the Syro-Palestinian coastal area is in fact particularly suitable, as it contained the right percentage of calcium carbonate, in the form of tiny shell fragments[9,48,70,87,101], specially the Belus River sands[102]. It is believed that the sand was collected in this region but the actual process of glass-making occurred[48] in factories located to the west and east.

A limited number of primary production centres found in Egypt and Syro-Palestine were dated to Late Roman and Byzantine period, but for earlier Hellenistic and Roman period, no primary furnaces have been reported. Aside from sand sources in Syro-Palestinian area, Roman historians also suggested sands from Italy (near the Volturno River), Spain and France. A recent study found that some western Mediterranean sand deposits could be suitable for Roman glass production. Six limited areas, including Basilicata, Puglia and Tuscany in Italy, Huelva province and Murcia regions in Spain and from the Provence in France. The last three areas would have produce a composition similar to Roman natron glass after the addition of an extra source of lime[103].

3.3.3.3 Additives

The appearance of primary natron glass could be changed using additives, usually metal-containing minerals,in order to opacify, colour or decolour glass. The additives could have been added in both the primary or secondary workshops as well. There are two most common opacifiers found in antique glass. Lead antimonate ($Pb_2Sb_2O_7$) found in green and yellow opaque glass and calcium antimonate ($Ca_2Sb_2O_7$) in white opaque glass. They could had been added in a mineralogical form as bindheimite or stibnite or by adding litharge a residue from the silver and gold refining process. Between the 2nd and 4th century AD there was a change from antimony to tin based compounds[104]. The decolourizers found in Roman glass are manganese dioxide (MnO_2), antimony pentoxide Sb_2O_5 or a combination of both. Only antimony or antimony with more than 1% of manganese is found in colourless glass, whereas less of 1% of Mn can be found in colourless ones but with a shade of blue-green glass up to green-blue colour, although the colour can not always be related entirely to the decolourant but also to the firing conditions[45,105].

As for colouring, the most colouring agents found are Fe^{2+}, Fe^{3+}, Cu^+, Cu^{2+}, Mn^{3+} and Co^{2+} ions[1,62]. Iron oxide would be added as crocus martis or Crocumferri and sometimes as haematite (Lapis-ematitis) causing, depending on its oxidation state a distinctive blue or reddish colour[96]. Copper was added though burning metal copper scales to obtain red and the black oxides, Cu_2O and CuO, respectively. Red copper oxide was used to make ruby-red glass and black copper oxide was used to make green and aquamarine glasses[25]. Manganese dioxide was mainly used as a decolourant, in quantities ranging from about 0.3 to 0.8 % to counteract the colour given by iron. Lastly in blue colour glass the main colourant found would be copper, cobalt or iron and would be discussed in more detail in the next section[1,25,61].

3.4   Blue Roman Glass

3.4.1   Matrix composition

Around the mid-1st century glass had become much less expensive than before[106], and the use of strongly-coloured vessel glass declined against the colourless or slightly coloured glass[70]. The centralised model of glass production has also been suggested for highly coloured glasses from the 1st century considering that different glass colours exhibited different composition, not entirely explained by the mineral additives added for colouring or opacifying, but for their main composition[107,108].

For example, blue, purple, colourless and amber glasses would typically be natron glass, while translucent emerald green and opaque red glasses (both coloured with copper), would diverge from other glasses of this period and later, presenting different levels of potassium and sodium, suggesting plant ashes as flux (early 1st millennium production traditions). This can be supported specially for blue glass

due to evidence of blue-coloured glass chunks from the 1st century found around the Mediterranean[9], they were likely to have been produced at a primary location (where it was coloured) in a much earlier time (see Pulak[10]). As for the most prevalent colour through the 2nd to the 4th centuries record, blue and green colours, Sanderson et al.[6], and Jackson et al.[109] showed that Roman glasses were not compositionally homogeneous, and that some strong groups of late Roman, and early post-Roman as well as the influence of recycling can be reflected in the composition.

3.4.2 Blue Colour

For highly- coloured blue glass it is known that Romans used copper, cobalt, ferrous iron (within a reductive atmosphere in the furnace) or a combination of the previous compounds to produce different hues in glass. Copper oxide produces a pale blue colour with at least 1–2% CuO while iron oxides can produce a wide variety, not only depending on the oxidation state, but also on the quantity and presence of other oxides (manganese oxides). Concerning highly coloured, deep blue, cobalt oxide in a concentration as low as 0.01% can produce the desired colour.[5,25,61,110] (see figure 6.1)

Cobalt can naturally occur as sulphides ($CoAsS$, Co_3S_4), as an impurity of manganese, iron, copper and silver ores as asbolane or arsenides deposits. Asbolane formula is $Mn^{4+}(O,OH)_2 \cdot (Co,Ni,Mg,Ca)_x \cdot (OH)_{2x} \cdot nH_2O$ with impurities of Al, Ca, Fe, Si. Arsenides ($CoAs_{2-3}$) on the other hand are usually associated with iron, copper or silver ores[5]. Due to this consistency, using trace elements for determining the production location and period of dark blue glass beads, within Near Eastern, Mediterranean and Europe, from the Bronze Age to the 18th century AD, has been proven effective. Cobalt, used as the main colouring agent can be associated with arsenic, nickel and bismuth[111]. Particularly from the beginning of Iron Age, to the 12th century in Europe, cobalt is related to Sb, Cu and Mn, probably coming from the Near East and closely associated with natron soda-lime glass.[111].

Before the 12th century AD, the cobalt production has been so far related to two regions, the area Kashan in Persia and the Erzgebirge region in Saxony, Germany[7]. In the Erzgebirge mountain range, used to be mined for silver, with particular impurities (Ni–Co–As–Ag–Bi)[112]. Studies on slags from the region showed high concentrations of the heavy metals Pb, Cu, Zn and U as well as As, the main composition of the slags was similar, but the concentrations in Ba, Ca and partucularly U, could change greatly from sample to sample[113]. The cobalt-rich alum deposits of the oases in the western Egyptian desert, show elevated levels of nickel, zinc, manganese as well as alumina, magnesia and iron and a Pb-Sb-Sn-Zn relationship[5,98].

During the first millennium AD it is known that cobalt sources were of high purity compared to the first millennium BCE, making it harder to identify the origin of the source. Only the higher concentration of iron and copper, or some other trace elements like nickel, manganese, zinc, arsenic and lead will distinguish blue glass

(a) Free-blowing glass *unguentarium* from Aosta (early 1st century AD). Regional Archaeological Museum, Aosta, Italy.

(b) Mold-blowing bottle, from Camarina (end of 2nd to 3rd century AD). Regional Archaeological Museum of Camarina, Ragusa, Italy.

(c) Glass beads necklace, from Sofiana (4th-5th century AD). Regional Archaeological Museum of Gela, Caltanisetta, Italy.

(d) Translucent bi-handled urn with decorative zigzag motif in blue (4th century AD). Museu d'Arqueologia de Catalunya, Barcelona.

Figure 3.2. Examples of blue Roman glass. (photos fromMollo and Framarin[77]

from other coloured and uncoloured, but these elements would probably be more related to the raw materials than to the colourant[5].

Gratuze et al.[5], on a study of a 2nd to 3rd centuries glass workshop in France, calculated the average composition of the colourless raw glass (antimony), and subtracted from the average composition of the blue glass, in order to single out the elements that could be associated with cobalt colourant, giving: iron, cobalt, nickel, copper, tin and lead and in smaller proportion gallium, indium and gold. Particularly, a ratio of CoO/NiO between 24 and 54 demonstrated to describe (along with studies of more workshops from the same time) the source of cobalt colourant used during the 2nd to 4th century AD. It was noted that CoO/NiO ratio can variate when there is low cobalt concentrations due elevated heavy element levels.

3.5   Recycling

Although it is known through Roman literature about the recycling and reuse of glass[114], inside archaeological contexts the evidence is not always clear[70,115,116]. From several archeological records, meaning, glass chunks, cullets, and furnaces, it seems like this kind of glass were mostly low-status-vessels[117,118] and windows[115]. Recycling would most likely occur close to urban areas[87], but saying more about the process is complex because it could depend on the context (e.g. Roman military vs a family)[116].

Some elements found in light coloured glass, like cobalt in a higher content than the usual impurities in glass-making sand can indicate that high coloured glass was used in the recycled batch[119]. This is the same case of iron when found in high levels, it is believed that it could have been added as a contaminant from the workshops. Inside the workshop some other elements due to contamination could be K,Mg and P due to ash. Some other effects observed could include the loss of alkali fluxes (specially sodium) due to progressive reheating, dirt inside the batch and a visible deterioration of the colour[115].

Romans used MnO_2 and Sb_2O_5 as decolourants that could be added in the primary or secondary workshops. Some glass analysis from Roman primary workshops, or within shipwrecks have shown the presence of either manganese or antimony on primary glass or glass found close to the primary workshops[89,120,121] so finding both within the composition could be a clear evidence of recycling[117,122]. As recycling can affect compositional groups[123], isotopic ratios, like $^{87}Sr/^{86}Sr$ have proven to be effective because they have little or no change under re-melting and recycling[1,124]. It has been proved that it is possible recognize an intermediate composition when two types of glasses are mixed because the group will be drawn between the "parent" types.[115]

Chapter 4

Archaeological Site: North-East Palatine Hill

4.1 Location

Since 1986, the Chair of Methodology and Techniques of Archaeological Investigation has been excavating the valley of the Colosseum[125]. In 2001 the excavations of the north-eastern slope of the Palatine started. The slope runs alongside the substructures of the *Vigna Barberini*, from the Arch of Constantine to the Arch of Titus (see figure 4.1) inside the current Archaeological Park of the Roman Forum-Palatine Hill.
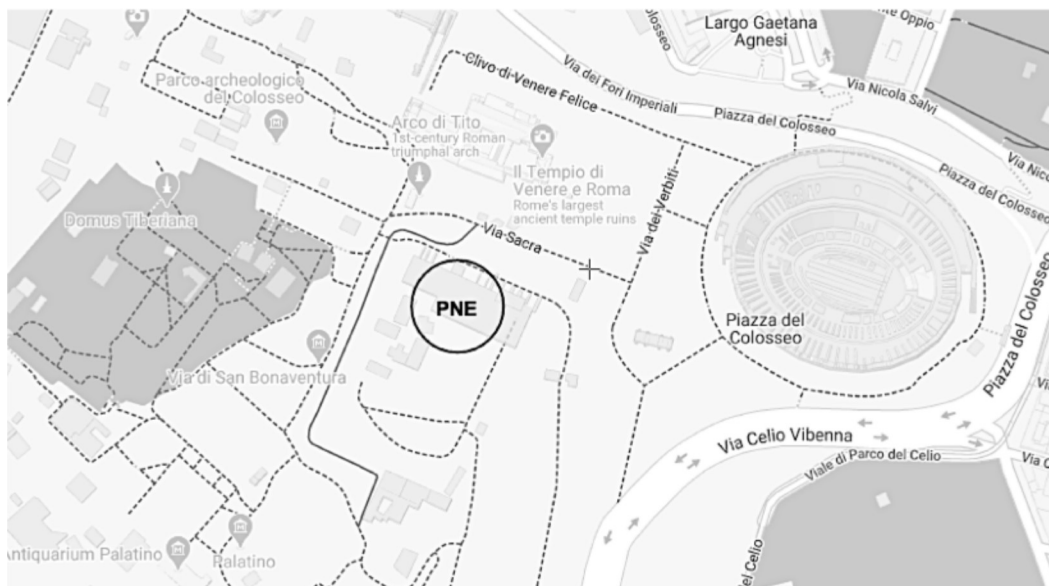

Figure 4.1.   Detailed map of the centre of Rome with the location of the Palatine North-East (PNE) site. ©Google

The Palatine North-East site located at located 41°53'24"N 12°29'20"E is the complex known as Baths of *Helagabalus*, situated on the north-east slope of the Palatine Hill[126]. The site is divided from the remains of a monumental Flavian fountain (*Meta Sudans*) by the pedestrian road that goes up to the Palatine, but the stratigraphies found show an environmental and topographical continuum[127]. According to the geomorphology, before its occupation the landscape of the whole area was formed by steep slopes and watercourses in the bottom of the valley[128].

4.2   History and Archaeology

Research has pointed out different "key" moments of human occupation recorded in the urban history of Rome city centre separated in two phases[128]: before and after the fire of 64 AD. The earliest remains of features are from the Iron Age (end of the 11th to the 8th century BC)[127]. The story continues with the foundation of the city in the 8th century BC, goes through the Roman Kingdom, Republican and Imperial periods and finishes with medieval and modern remains before the 19th century. To begin with the description of the archeological site, at the end of the 8th century BC there are solid earthworks of the two roads directed from the now *Circus Maximus* to the Esquiline, and from the valley (that will later be of the Colosseum) to the Roman Forum. For the late 7th-early 6th century BC, indirect traces of a sanctuary located on the southern slope of the Velia Hill, and remains of a second sanctuary located on the northeastern corner of the Palatine Hill (probably the so-called *Curiae Veteres*).

The two centuries of the Archaic age (mid-6th and 5th century BC) are characterized by intense construction activity, monumentalization of places of worship, the arrangement of infrastructures (canalizations, substructures, roads, sewage system) and the appearance of the first stone buildings. The growth of a residential building (6th century BC) gradually occupied the valley and the surrounding hills and then spread along the two ancient paths on the edge of the hills[128].

Later, during the Republican age (5th-1st century BC), the intensification of urbanization required a clear distinction between the blocks intended for the sacred activities and those reserved for residence[127]. Infrastructures were renewed and private buildings began to have a certain character. In the mid-2nd century BC, there was a development of a room complex (*Domus*) in the valley along the direct route to *Esqulino*.

Throughout the Augustan and Julio-Claudian ages, the site saw the erection and restoration of monumental and symbolic architecture (like *Meta Sudans* and Claudio's temple in the ancient Palatine sanctuary). In the Augustan age, the extensive and structured building activity is documented by the continuous refections of the road paving, underlying sewers (in cement, travertine and brick), the recurring restoration of *Domus*, and by the remains from structures (first as reticulated work marks and then in brick). The most important finds are recorded in this sacred area and in

front of it in the valley.

The fence of the *Curiae Veteres* is newly rebuilt in brick and houses an audience and a large travertine staircase towards the Palatine. In front of the entrance to the *Curiae*, an ornamental construction on the intersection of the two previously mentioned streets, maybe the first *Meta Sudans* from Augustus age (16 meters high and with a rectangular basin animated on the long sides by an exedra), was present. During the Claudian Age, between 51 and 54 AD, due to a fire, there was a reconstruction of a burnt temple, and the *Meta* was completely rebuilt. This first long phase ended with the Neronian fire of 64 AD.

The urban layout practically did not change until the fire, which destroyed, according to ancient writers, the Circus Maximus and a big part of the city. On top of the ruins, Nero imposed a new urban plan in the valley and the surrounding hills of the new imperial residence known as the *Domus Aurea*. The *Meta Augustea*, with the crossroads and the two ancient sanctuaries, disappeared. The direct way to the Forum remained, but with a different height and orientation than the ancient track up the hill that was used for eight centuries. This new axis is still traced today by the current *Via Sacra*.

The excavation returned impressive traces of the fire and made it possible to identify: the remainings of the *Meta Sudans* after the stripping of all its valuable or reusable material; a rise of the walkways of more than 4 meters by discharges of building debris and, towards the Palatine, the cutting of the slope that stops at the height of the late-Republican/Augustan *Domus*. The building blocks of the *Domus Aurea* arranged along the roads towards the Esquiline and the one leading to the Forum were rectified and decorated with arcades. The buildings consisted of a system of porticos and terraces built around a *stagnum* (in the area later occupied by the Colosseum).

Therefore, the second phase started with the construction of the Neronian *Domus Aurea*, continues with the extensive constructions of the Flavian and Hadrianic ages, and ends with the Severan (III century AD), Massenzian and Constantinian building activities. The last key moment, after which there will no longer be any radical changes in the area, happened during the Flavian interventions. Immediately after Nero's death in 68 AD, the area returned to its public functions, some structures of the palace built up to that moment were broken down, the Colosseum was built in the place of the lake, and this created a functional square for the entertainment[128].

Nero's death interrupts the construction of the *Domus Aurea*. The Flavian emperors (69-96 AD) completed the unfinished works and they equally created colossal structures (like the Amphitheatre instead of the *stagnum* and the Arch of Titus). They restored some pre-existing monuments destroyed by fire (the *Curiae*, the *Meta* and the temple of Claudio in the *Celio* district), but did not modify the Neronian urban layout. In the excavation belonging to this period they found the rebuilding of (what would become in the Neronian era) the new intersection between a road to the Esquiline and that towards the *Meta Sudans Forum* (18 meters height

and the circular tank of 16 meters)

During the Hadrianic age (117-138 AD) a new urban project was implemented. The construction of the Temple of Venus and Rome on the structures of the Neronian vestibule on the Velia Hill, the rearrangement of a terrace (now known as *Vigna Barberini*) on the Palatine Hill, the expansion of the *Meta Flavia* basin and the construction of the *Arco di Costantino* increased the monumental impact of the site, which remained unchanged until the 5th century[128].

In 191-192 AD during the Severan age (192-235 AD) a new fire struck the Forum and probably damaged the north-eastern slope of the Palatine. A temple framed by arcades was built on it at the beginning of the third century, and a few years later it was dedicated by Helagabalus to the god of Emesa. The Hadrianean *horreum* was also demolished and replaced by a new building two-storey *horreum* in brickwork, with rooms arranged around a large courtyard that entirely occupied the space between the road towards the Forum and the substructures of *Vigna Barberini*. As for the Massenzian age (306-312 AD), the recovery of the entire southern slope of the Velia from the *via Sacra* until valley of the amphitheater (including the Temple of Romulus, the Basilica and Temple of Venus and Rome), the expansion of the *Meta Sudans* basin, and perhaps intervention on the base of the Colosseum had a significant impact on the organization of the spaces in this urban sector[127].

The excavation for this period showed the foundation pit of the Arch of Constantine (315 AD), the insertion in the Severan *horreum* of a new structure in work (the courtyard paved with white marble tiles, fountains, flower beds, tubs with marble coating) and, towards the Arch of Titus, an apsidal room that opens onto a space occupied by a basin paved with white marble slabs, flanked by two other *triclinium* rooms and a small *balneum* built behind this ensemble (hence the name *Terme di Elagabalo* given in the history of studies to the entire complex). Here, however, the definitive "crisis" of the Late Antiquity complex seems to appear later (between the 6th and 7th centuries), than in the block of the ancient *Curiae*. As for the rest of the area, by the second half of the fourth century the settlement coming to dissolve and the presence of emperors and the imperial court in the Palace was rare. Modest maintenance works constructions of the Palatine were found.

In the second half of the 5th century AD, to avoid the collapse of the Neronian-Flavian terrace, the basement compartments of the building were filled up to the vaults with earth drains so that the upper floors have still accessibility. There is not much information about the function of the masonry building overlapping the Neronian/Flavian semi-hypogeal rooms, mostly buried with discharges of debris and rubble at that time because the preserved structures are fragmented. Some semi-hypogeal rooms were abandoned at the end of the century[128].

Between the 5th and 6th century AD, the installation of a metal workshop took place in the rooms south of the Flavian temple, in one of the colonnaded areas close to *Piazza dell'Anfiteatro*[128]. It was probably in use till the 6th century. There is a repaving with marble for reuse of the *triclinium* rooms of the *stibadium* (first

half of the 6th century AD) and an insertion in the courtyard of a new *apsidal* structure (6th century AD), whose walls contain in the foundation, reused materials like high-quality marble from imperial portraits (Septimius, Severus, principles of the Severan and late-Severan dynasty), herms and busts. Finally, the abandonment of the sewage system in the valley and the Palatine was followed by the consequent filling of the pipes themselves (5th-6th century AD)[127].

In the early Middle Age, findings of necropolises and spoliation marked the abandonment of this part of the ancient city, which later saw the construction of small churches and an inhabited area near the Arch of Constantine. The final recovery of the area takes place with huge earthworks and restorations to the Arch itself and to the *Meta Sudans* during the nineteenth century. Between 1931 and 1936 the area acquired its current appearance, with the demolition of the remains of the *Meta* and base of the Colosseum and the construction of the driveway square.[127]

In summary, the Palatine North-East site , currently known as Baths of Helagabalus, has been continuously changing, from the end of the Iron Age to present times. After witnessing the foundation of the city in the 8th century BC,t it experience the creation of impressive infrastructures, and during Imperial age, it supported the construction of two large series of utilitarian rooms (*horreum*), with front to road connecting the Colosseum with the Forum, and after the severan period (end of the second-early third century AD), an new and different structure, with rooms arranged around a central rectangular courtyard. The collection of blue glass samples belongs to different stratigraphic layers from contexts dated from Tiberian age (14-37 AD) and until the last quarter of the 5th century AD. The context dates presented come from the study of materials and stratigraphy made by the archaeologists who are still working on this project, thus they are not published in detail.

Chapter 5

Materials and Methods

5.1 Glass Samples
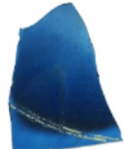
Twenty six samples from the North East Pallatine archaeological site, specifically from the complex known as Baths of *Helagabalus* were studied. The selection of only blue samples, belonging to the 1st to 5th centuries was performed by the archaeologist in charge of the material Barbara Lepri. The collection of blue glass, comprises examples from contexts dated from the 1st to the 5th century. Most pieces are shards of different functional objects like beakers, Skyphos, flasks, bottles and some particular pieces like a decoration plaque and chunk of raw glass, all in high-coloured blue or with high-coloured blue decorations.

The samples, aside from the context period, were dated based on parallels and typologies. The chronology of the samples starts approximately in the second half of the 1st century BC. Ten of the samples belong to this time period and until the 1st century AD. There are all containers like bowls, flasks, and a beaker, most probably moulded through casting, there is one example of an *unguentarium* made by glassblowing and a decoration panel made by hot forming and cutting.

From the 2nd century onward the glass making technique identified for the samples that belong to containers is blown glass. The samples are splinters of containers like beakers, plates, bottles, and flasks dated from the 2nd to the 5th but all found in a 5th century context, probably ready to recycle or being reused. Aside from pieces of containers, there is a mosaic tessera from the imperial Roman age made by hot forming and cold cutting. Finally the collection has two interesting pieces of raw glass, one properly moulded as an ingot and a chunk, both belonging to two late antiquity periods.

In the next Table (5.1) the complete details with the photographs attached can be seen.

Table 5.1. Samples with archaeological context.

Sample No.	Sample	Object	Technique	Context date	Glass date	Typology
V1		Bowl rim	Casting	Tiberian	Second half/late 1st c. BC-early 1st c. AD	Grose 1989, p. 247, Linear-cut bowl
V02		Bowl wall	Casting	Flavian	Second half/late 1st c. BC-early 1st c. AD	Grose 1989, p. 247, Linear-cut bowl
V03		Bowl rim	Casting	Tiberian	Second half/late 1st c. BC-early 1st c. AD	Grose 1989, p. 247, Linear-cut bowl ?
V04		Bowl wall	Casting	Neronian	1st c. AD (prob. First half)	Isings 3
V05		Bowl wall	Casting	Hadrianic	1st c. AD (prob. First half)	Isings 3

Table 5.1 continued from previous page

Sample No.	Sample	Object	Technique	Context date	Glass date	Typology
V06		Stelo modanato of (probably) a Skyphos/kantharos/goblet	Casting	Tiberian	First half of the 1st c. AD	Gorga 30, var. 2 (Gruppo Grose 1989, Family III, pp. 254-256)
V07		Kantharos/bell-shaped beaker rim	Casting	Tiberian	First half of the 1st c. AD	Gorga 14-15 (Gruppo Grose 1989, Family III, pp. 254-256)
V08		Bowl rim	Casting	Flavian	Second half/late 1st c. BC-early 1st c. AD	Foy 2005, fig. 17, n. 1 (Linear-cut bowl)
V09		Unguentarium wall	Glassblowing	Tiberian	1st c. AD	Isings 6

Table 5.1 continued from previous page

Sample No.	Sample	Object	Technique	Context date	Glass date	Typology
V10		Plaque for architectural decoration fragment	Hot forming and cutting	Flavian	1st c. AD	Grose 1989, n. 671
V11		Bowl/dish ring base (solid)	Casting	Second half of the 6th c. AD	Late 1st/early 2nd-3rd c. AD	Gruppo Grose 1991
V12		Beaker coil base	Glassblowing	Last quarter of the 5th c. AD	Late 3rd-4th c. AD	Isings 106/109?
V13		Beaker coil base	Glassblowing	Last quarter of the 5th c. AD	4th-early 5th c. AD	Isings 106/109?
V14		Plate? rim (tubular)	Glassblowing	Second half of th 6th c. AD	-	-

Table 5.1 continued from previous page

Sample No.	Sample	Object	Technique	Context date	Glass date	Typology
V15		Bottle handle	Glassblowing	Last quarter of the 5th c. AD	Late 2nd/early 3rd-4th/early 5th c. AD	Isings 126/127
V16		Bottle handle	Glassblowing	Last quarter of the 5th c. AD	Late 2nd/early 3rd-4th/early 5th c. AD	Isings 126/127
V17		Flask rim	Glassblowing	Last quarter of the 5th c. AD	5th c. AD	Sternini 2001, fig. 15, nn. 147-148
V18		Flask rim	Glassblowing	Last quarter of the 5th c. AD	5th c. AD	Sternini 2001, fig. 15, nn. 147-148
V19		Flask? high ring base	Glassblowing	Last quarter of the 5th c. AD	4th-early 5th c. AD	

Table 5.1 continued from previous page

Sample No.	Sample	Object	Technique	Context date	Glass date	Typology
V20		Bowl rim (coil applied)	Glassblowing	Last quarter of the 5th c. AD	4th–early 5th c. AD	Sternini 2001, fig. 13, n. 128
V21		Bowl rim (tubular)	Glassblowing	Last quarter of the 5th c. AD	4th–early 5th c. AD	Sternini 2001, fig. 7, n. 42
V22		Cup/beaker wall with a blue blob	Glassblowing	Last quarter of the 5th c. AD	Late 3rd–4th c. AD	Isings 96/106/109
V23		Bowl/lamp rim (tubular) with an horizontal handle	Glassblowing	Last quarter of the 5th c. AD	4th–early 5th c. AD	Uboldi I. 4? Lepri, Magyar in c. s. , Pl. 2, no. 51 (Sternini 2001, fig. 7. 48-49 senza anse)
V24		Mosaic tessera	Hot forming and cold cutting	Last quarter of the 5th c. AD	Roman imperial	

Table 5.1 continued from previous page

Sample No.	Sample	Object	Technique	Context date	Glass date	Typology
V25		Raw glass (Chunk from an ingot)	Primary production	Last quarter of the 5th c. AD	Late antique	
V26		Raw glass (Chunk)	Primary production	Last quarter of the 5th c. AD	Late antique	

Gorga = A. Petrianni, Il vasellame a matrice della prima età imperiale, Collezione Gorga, Vetri I, Firenze, 2003

Grose 1989 = D. F. Grose, Early Ancient Glass, The Toledo Museum of Art, New York 1989.

Lepri, Magyar in c.s. = B. Lepri, Z. Magyar, "The glass", in M. Lavan, M. Mulryan (eds.), Public Space in Late Antique Ostia: Excavations and Survey 2008–2012, in press

Isings = C. Isings, Roman Glass from Dated Finds, Groningen-Djakarta 1957

Sternini 2001 = M. Sternini, "Reperti in vetro da un deposito tardo antico sul colle Palatino", in JGS 43, 2001, 21-75.

Uboldi = M. Uboldi, Diffusione delle lampade vitree in età tardo antica e altomedievale: spunti per una tipologia, in 'Archeologia Medievale', XXII, 1995, 93-145
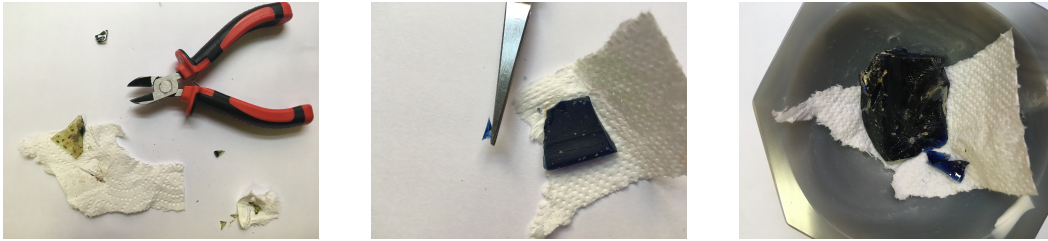
Figure 5.1. Tools used to break the archaeological samples: tweezers, scissors and mortar.

5.2 Sample Preparation

Due to the nature of the analytical techniques employed, sample preparation was required. Each glass shard was carefully broken with different tools to produce a smaller piece, trying to minimize the damage on the original as much as possible (fig. 5.1).

The glass splinters were carefully placed on a pre-prepared base with circular moulds (fig. 5.2a). The HARDROCK 554 epoxy resin was weighted, mixed and then poured inside the sample moulds (fig. 5.2b). The epoxy resin pills were left to dry and then carefully polished to expose the glass.
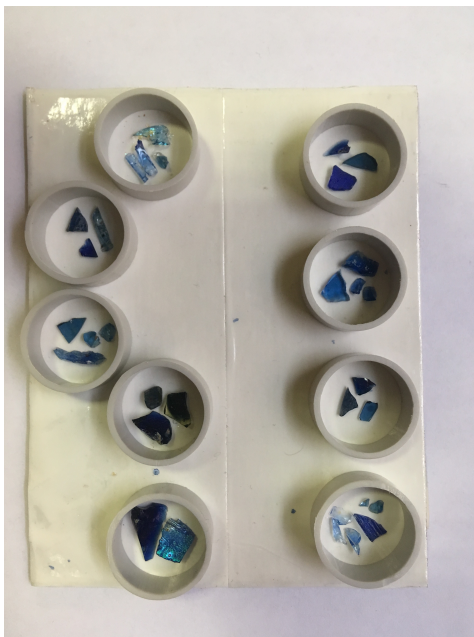
(a) Nine pill moulds with the mounted samples.

(b) Pouring the epoxy resin inside the mounted samples.

Figure 5.2. Sample preparation procedure.