A CHEMICAL COMPARISON OF BLACK GLASS SEED BEADS FROM NORTH AMERICA AND EUROPE

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Analysis of the elemental composition of glass has gained traction over the past few decades. The growing interest and utilization of non-destructive and micro-destructive analytical techniques has allowed for a more in-depth understanding of glass production, distribution, and consumption. The analysis of glass trade beads in particular has led to the development of a chronological sequencing for non-diagnostic seed beads opacified with metal oxides as well as ore sourcing for cobalt-blue and red beads. There is deficient research on 18th-century glass bead composition, especially of black glass beads. This article explores the elemental composition of 149 black seed beads from three 18th-century sites in Pensacola, Florida, and compares the assemblage to a small sample of similar glass beads (N=11) recovered from two sites in the United States as well as three potential glass production centers in Europe.

INTRODUCTION

Glass beads were a major commodity for Native Peoples and are ubiquitous at European and Native archaeological sites in the southeastern United States. Analysis of glass bead assemblages has been used by archaeologists to construct basic temporal sequences, as well as to interpret aspects of Indigenous sites, such as traditions of adornment, value systems, social standing, exchange, group and personal identity, consumption, daily practice, and the nature of colonial entanglements (Francis 1988:292; Walthall 2015:259).

Among the great variety of glass bead forms, drawn glass seed beads are the most common and abundant. They are of either simple, compound, or complex construction and generally less than 4 mm in diameter, making them ideal for sewing onto clothing and other personal items or worn as adornment in the form of necklaces, wristlets, or anklets (Avery 2008:57; Blair 2015:91; Deagan 2002:131). Unfortunately, glass seed beads offer no physical diagnostic features to accurately assign them an origin of manufacture. Moreover, the common production of simple seed beads throughout Europe over a vast time span does not allow for much interpretive insight into the trade and distribution of this bead form.

In recent years, research has shifted towards not only looking at the structure, manufacture, and morphology of beads, but also analyzing their chemical composition, opening avenues of inquiry into various aspects of their production and consumption that would normally be unattainable through physical analysis alone. These techniques can even give physically undiagnostic beads (like seed beads) much more data potential and could even evidence regional distribution based on the identification of compositional groups both within and between coeval archaeological sites. In terms of chemical variability, the wide distribution of glass trade beads makes them a useful indicator of participation in specific trade networks (Walder 2013:120).

The characterization and patterning of primary glass ingredients can also be used to identify the place of manufacture, the source of raw materials, and the evolution of glass recipes used (Blair 2017:32). The chemical composition of glass is an important source of information about the provenience of a single object, but it can also support knowledge about the technological history of glassmaking obtained from the technical literature and other historical documents in archives and libraries (Wagner et al. 2008:415). In recent studies, element chemistries of glass beads were used to sort beads into groups, using elemental concentration fingerprints (Karklins et al. 2001:188). These fingerprints can relate to glass recipes because they reflect not only batch composition, type of applied raw materials, or their source and method of preparation, but also the various technological conditions of glass production (Wagner et al. 2008:416). Additionally, ingredients and their shifting ratios to one another can function as temporal markers (Walder 2013:138).

In general, laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) is extremely useful for

analyzing any vitreous material (glazes and glasses). An abundance of chemical research has been conducted on white (Blair 2017; Hancock, Aufreiter, and Kenyon 1997), red (Sempowski et al. 2001), blue/turquoise (Hancock, Chafe, and Kenyon 1994; Walder 2018), and other colored beads (Burgess and Dussubieux 2007) recovered from archaeological sites in the United States and elsewhere using LA-ICP-MS and other methods like portable X-ray fluorescence (pXRF) and instrumental neutron activation analysis (INAA). There is, however, a dearth of information concerning the chemical composition of black glass, with the exception of a pXRF study by Robert B. Templin III (2017).

THE COMPOSITION OF BLACK GLASS

Common colorants in glass are transition metal compounds of iron, lead, tin, copper, and cobalt (Dussubieux 2009:101). In most cases, black glass is saturated with blue, green, brown, or violet pigment that gives it the appearance of opaque black (Dussubieux and Gratuze 2012). Violet glasses contain on average 1% manganese, but dark-glass samples have high concentrations of manganese ranging from 3.4% to 13% (Dussubieux 2009). In general, glasses high in manganese are also high in strontium, slightly elevated in barium, and lower in chromium and vanadium (Bertini et al. 2011). Barium is found in barium-manganese ores associated with pyrolusite which was used in glassmaking in the 17th century. The relationship between manganese and barium may also help identify the geographical region where the ore was sourced (Templin 2017). Another way to create black glass is to use nickel with cobalt, which also opacifies the glass. Adding nickel to heavily leaded glass or glass with potassium creates a violet to deep violet color, respectively (Weyl 1959). Iron concentrations are sometimes high in black glass especially for dark green or brown (Veritá and Zecchin 2008:112). For dark green, a raw tartar (potassium tartrate) decomposer is present in the glass melt and also acts as a reducing agent.

In addition to metal oxide colorants, several elements were used to opacify glass, including tin, antimony, arsenic, and lead. Using the chemical analysis of white glass beads from relatively well dated archaeological sites of the 17th-19th-centuries, researchers have been able to establish time periods during which the opacifying elements tin, antimony, and arsenic were used successively (Blair 2017; Hancock, Aufreiter, and Kenyon 1997; Hancock et al. 1999; Sempowski et al. 2000). Research suggests that early 17th-century tin-rich drawn beads were replaced sometime later in that century by antimony-rich beads, and this pattern is emulated in glass workshops all over Europe signifying an economic reason for the shift. This argument has been strengthened by two studies of opacifying agents used in opaque white and black glass beads from a 17th-century Spanish site (Blair 2017; Templin 2017). The use of colorants and opacifiers facilitates the identification of chemical groups and subgroups within glass samples. Finer chemical groupings could come from a single batch of glass or from batches of glass made with similar proportions of ingredients, over a short period of time (Kenyon, Hancock, and Aufreiter 1995:329; Sempowski et al. 2001:513). Although little is known about black glass recipes, there is growing knowledge of the nature of bead production in major beadmaking centers in Europe.

THE SAMPLE SITES AND THEIR BEADS

The United States

During the 18th century, three presidios (fortified towns) were established as outposts of New Spain to protect the western extent of Spanish Florida from French and British encroachment and housed Spanish soldiers and residents. By the 1740s, two missions had been established for the Spanish-allied Apalachee and Yamasee Native groups living in the region (Figure 1). The Spanish missions of San Antonio de Punta Rasa and San Joseph de Escambe were both peripheral to direct Spanish control and proximal to French and British settlements. Their locations allowed them to act as trading hubs that moved supplies indirectly into presidios via access to Upper and Lower Creek trade networks. The Creek Native factions allied with the French, British, and Spanish depending on the benefits of the relationship. While the black glass beads from the Pensacola sites were made in various shapes using several production techniques, only drawn seed beads recovered from two presidios and one mission are reported here (Figure 2).

Presidio Santa Rosa de Isla (1722-1756) was the second iterative attempt by the Spanish to settle Pensacola Bay. Over 90% of the entire bead assemblage consists of drawn, monochrome, circular, heat-rounded seed beads. The most common colors are black, white (25.8%), and blue (24.2%). A sample of 41 black beads (Kidd and Kidd [2012] variety IIa7) was analyzed.

The occupation of Presidio de San Miguel de Panzacola (1740-1763) overlaps that of Santa Rosa. Over 80% of the entire bead assemblage consists of drawn, monochrome, seed beads. The principal colors are black (33%), white (24.2%), red (15.3%), and blue (12.5%). Thirty-eight black seed beads (IIa7) were sampled.

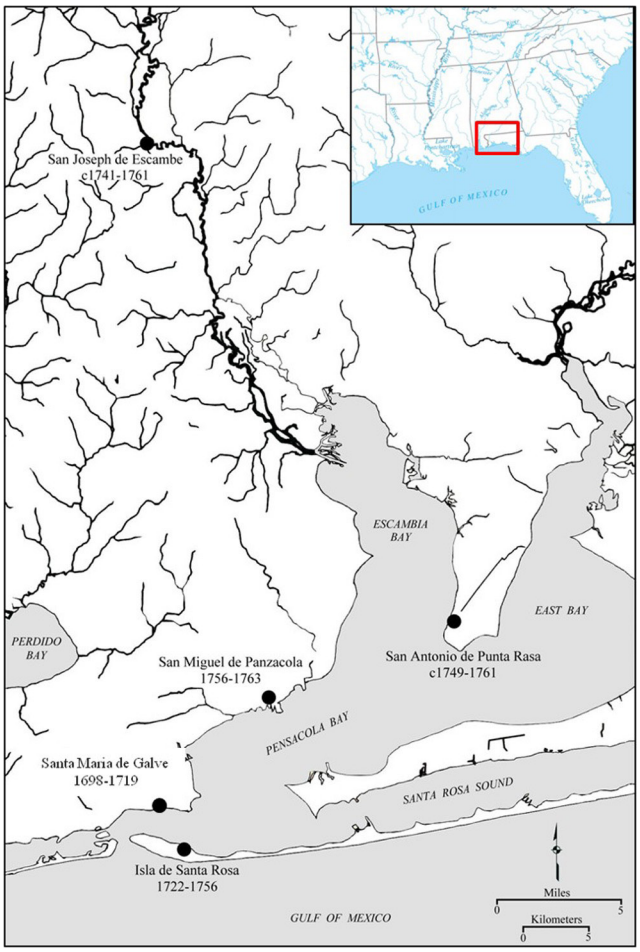

Figure 1. Locations of archaeological sites on Pensacola Bay, Florida (all images by author).

Mission San Joseph de Escambe was established upriver from San Miguel in 1741. Documentary sources imply that this was the new Apalachee mission under Chief Juan Marcos and lasted until 1761 when it was attacked by Creek raiders (Worth 2021). The entire glass bead assemblage (over 90%) is comprised of monochrome seed beads. Three colors dominate the assemblage and are equal in quantity: white (32.5%), blue (32.2%), and black (31.8%). Seventy black beads (IIa7) were sampled.

Chemical studies have also been conducted on drawn black beads from two late 17th-century French sites. Explorer Robert Cavelier Sieur de La Salle built a small fortification in the upper Illinois Valley in 1682 to establish a French foothold in the area (Walthall 2015). From 1680 to 1700, La Salle's mercantile system was the major source of goods, including glass beads, in the Illinois Country. The glass trade bead assemblage, dating to the 1682-1691 occupation of Fort St. Louis by La Salle's men, is dominated by very small and small monochrome beads in blue, white, and black (Walthall 2015:274). Walder (2015) sampled a black glass bead (IIa7) from Fort St. Louis during her dissertation research on opaque white and blue glass beads in the Upper Great Lakes region.

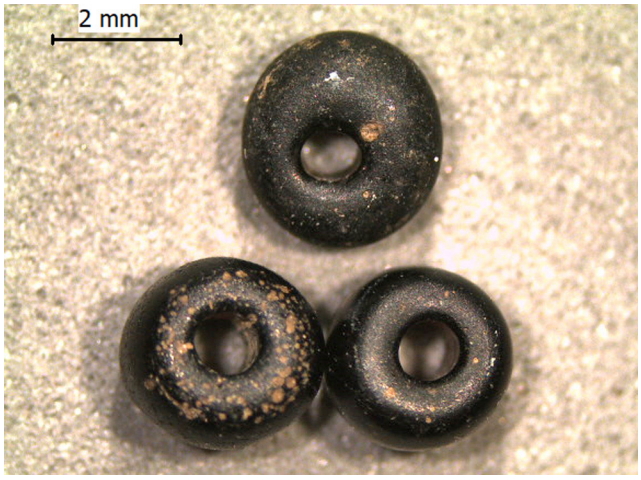

Figure 2. Black seed beads recovered from Pensacola sites (Prov #423-16).

Another archaeological site within the French sphere is the wreck of *La Belle*, located off the Texas coast. During La Salle's expedition to establish a colony and a shipping port at the mouth of the Mississippi River, *La Belle* became stranded and was abandoned in February 1686 in Matagorda Bay (Bruseth 2017). There are roughly equal amounts of white, blue, and black beads, with very small quantities of green, yellow, and red (Avery 2008:59). The shipwreck yielded over 200,000 black seed beads, most described as Variety 2 (small, circular, simple, opaque black, Kidd and Kidd IIa7). Three black beads were analyzed using LA-ICP-MS and their chemical compositions were averaged and presented in parts per million (Perttula and Glascock 2017:522). Additionally, Walder (2015:648) sampled five black beads (IIa7) from the Upper Great Lakes region. Since her sample is presented in wt. % and ppm for each individual bead, her data will be used for comparisons.

Europe

Three archeological sites in Europe that have yielded black or dark-colored beads provide comparative compositional data. All three sites date to the 17th century, but only two have ICP-MS chemical data suitable for comparative analysis. While INAA analysis was conducted on 11 black beads (IIa6 and IIa7) recovered from beadmaking wasters in Amsterdam at site Asd-Kg10, now attributed to the period from 1621 to 1657 (Hulst 2013:28), black glass is difficult to analyze using this technique since

the manganese isotope (^{56}Mn) degrades the sensitivities of calcium, cobalt, and tin (Karklins et al. 2002:119). Due to this fact, and that only 11 elements were recorded, this study was not included in the comparison. Three of the total glass samples chemically analyzed with ICP-MS in a later study were drawn black beads (IIa7) (Dussubieux and Karklins 2016:578).

Two sites in France (Espace du Palais and Cours Napoléon) also yielded black glass beads that were chemically analyzed. Glass wasters recovered from Espace du Palais came from a small workshop that used oil lamps to make various ornaments from glass rods or tubes produced by other specialized shops (Dussubieux 2009:97). Two of the six dark-glass samples are round or barrel-shaped drawn beads (Dussubieux 2009:99). Formerly living quarters, Cours Napoléon is located where the glass pyramid of the Louvre Museum now stands (Dussubieux and Gratuze 2012:26-27). Most of the beads are small monochrome drawn beads with black specimens being the most common, followed by turquoise, colorless, and dark blue. Sixty-three beads and wasters from this site (and one other not reported here) were sampled using ICP-MS to determine their place of origin (Dussubieux and Gratuze 2012:26-27). Of the total artifacts sampled, three were black drawn beads. Only the two beads from Espace du Palais have full chemical data reported for comparative analysis (Dussubieux 2009:103).

COMPOSITIONAL ANALYSIS

Methods and Materials

The chemical composition of the black beads was determined using a Teledyne CETAC Analyte Excite 193 nm excimer laser ablation system attached to a Thermo XSERIES 2 inductively coupled plasma mass spectrometer located in the Plasma Analytics Laboratory at the University of California Santa Cruz. Analysis was conducted by the author and analytical protocols and calculation methods were adapted from Gratuze (1999). The isotope ^{30}Si was employed as an internal standard, and the standard reference materials NIST610 and NIST612 were used for external standardization, along with Corning glass standards B (soda-lime-silica glass), C (lead-barium glass), and D (potash-lime-silica glass). Two spots of 110 µm were tested on each bead and averaged against a gas blank. Data were collected on 55 elements.[1] The results obtained for each artifact were normalized to 100% (Gratuze, Blet-Lemarquand, and Barrandon 2001). All trace elements are presented in parts per million (ppm) and accuracy ranges from 5% to 10%, depending on the elements and their concentrations.

Analysis Results and Comparisons

Very little comparable chemical data exist for black beads in the current literature. Of the data that are available, the analytical methods and/or elements reported differ greatly, making it difficult, if not impossible, to provide a full comparison of bead compositional groups. Additionally, sometimes only one bead was sampled from a particular site making any comparisons highly speculative and not statistically significant. Another substantial drawback is the lack of contemporary comparative samples. Most of the reported black-glass samples in the literature range from the Roman period to the 17th century. The following discussion represents a preliminary comparison of the Pensacola beads to similar colored beads from other sites in the United States and Europe.

Fluxes and Stabilizers

A total of 149 black beads were sampled from presidios Santa Rosa and San Miguel, and mission Escambe. The beads from the Pensacola sites cluster in one broad compositional group based on their fluxes and stabilizers (Table 1) (Figure 3). They have magnesium, potassium, and alumina levels higher than 1.5%, indicating a soda ash derived from halophytic plants rather than a mineral-soda or wood-ash source. Furthermore, both titanium and uranium are under 0.5%, and zirconium levels are under 100 ppm (most are under 50 ppm). In summary, the Pensacola beads contain silica most likely obtained from Spanish or Italian beaches, are fluxed with soda derived from littoral plant ash, and stabilized with calcium. This composition tentatively attributes their manufacture to Italy based on Venetian soda-lime-alumina glass recipes.

The black beads from San Miguel show less variation than those from Santa Rosa or Escambe. They contain less than 3% alumina, magnesium, and iron, whereas only a portion of the beads from Santa Rosa or Escambe show similar compositions (Figure 4). The San Miguel beads are well within the range of variation typical of Venetian soda-lime glass, but the tighter clustering indicates a single shipment of beads or perhaps a shift in the sand used to make the glass. The shift in materials could have become standardized by the 1740s, possibly indicating tighter trade restrictions for the Spanish presidio since the Escambe beads have more chemical variability in impurities associated with different silica sources.

The glass beads from *La Belle*, Espace du Palais, and Asd-Kg10 (with the exception of one bead) all cluster within the Pensacola bead assemblage, signifying a similar

Table 1. Means and Standard Deviations of Chemical Analyses.

Means	Santa Rosa	San Miguel	Escambe
Na_2O	10.69	10.06	10.79
MgO	2.42	1.98	2.51
Al_2O_3	2.52	1.82	2.67
SiO_2	61.74	64.78	62.82
P	0.65	0.75	0.62
K_2O	3.44	4.41	3.40
CaO	9.35	8.78	8.78
Mn	6.26	5.48	5.74
Fe_2O_3	1.53	1.17	1.62
Sb	0.43	0.11	0.20
PbO	0.41	0.19	0.31
n	**41**	**38**	**70**

Standard Deviations	Santa Rosa	San Miguel	Escambe
Na_2O	1.16	1.02	0.77
MgO	0.53	0.43	0.55
Al_2O_3	0.86	0.50	0.94
SiO_2	3.33	2.04	2.44
P	0.11	0.19	0.10
K_2O	0.92	0.77	0.90
CaO	1.02	1.13	1.33
Mn	2.89	1.90	1.59
Fe_2O_3	0.61	0.40	0.62
Sb	1.09	0.08	0.14
PbO	0.40	0.22	0.34
n	**41**	**38**	**70**

chemical composition of soda-lime-alumina glass (Figure 5). The beads from these sites also have similar amounts of iron, magnesium, and aluminum compared to the Pensacola beads, which further corroborates their shared production origins. A closer inspection of the black beads from *La Belle*, Fort St. Louis, and Asd/Kg10 reveals all have slightly elevated soda and lime content in comparison with the Pensacola beads (Figure 6). This variation could be a result of changes in recipes over time or obtaining the same raw materials but from different sources.

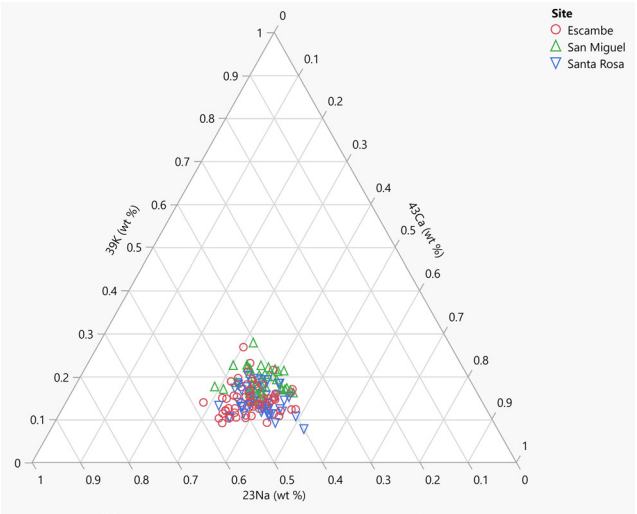

Figure 3. Ternary plot of flux and stabilizers in the glass beads from Pensacola (wt. %).

Colorants and Opacifiers

The concentration of manganese and/or cobalt oxides delineates four separate compositional glass groups within the Pensacola beads (Figure 7). Most of the black beads were colored with manganese (2%-11%) with under 100 ppm of cobalt. They comprise Group 1 which has a small sub-group in the beads from San Miguel that have high zinc concentrations (1000-1600 ppm) not associated with cobalt. Pyrolusite and other common manganese ores are not known to have zinc impurities, making this sub-group unique in its composition (Figure 8). The other manganese-colored beads have a positive correlation with barium with the San Miguel specimens having a slightly lower positive correlation (Figure 9). This strengthens the argument that

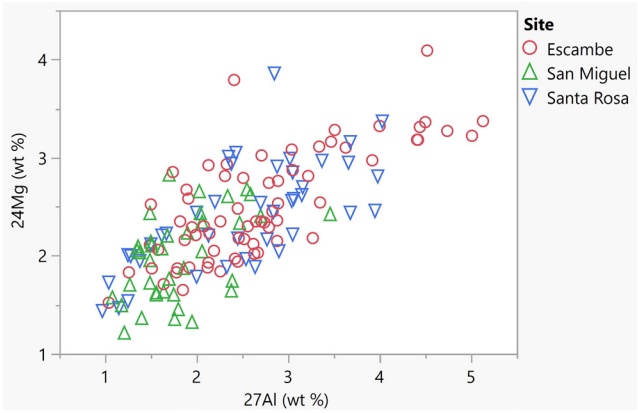

Figure 4. Biplot of manganese and alumina concentrations in the Pensacola glass (wt. %).

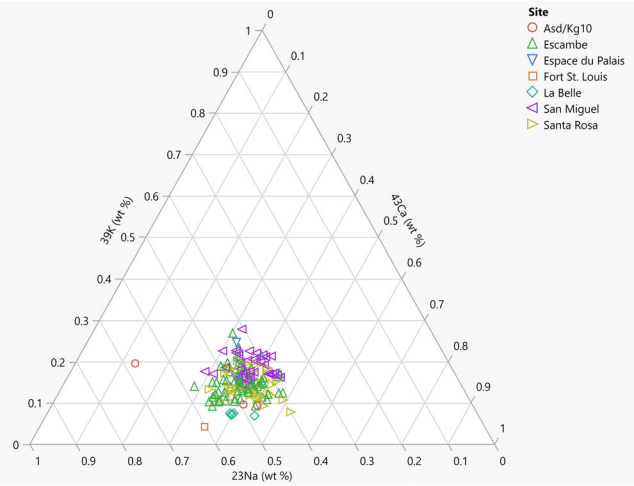

Figure 5. Ternary plot of flux and stabilizers used in the beads from all sample sites (wt. %).

the beads from San Miguel may represent a reduction in ingredient variance after 1740. The glass beads from *La Belle* site are compositionally similar to Group 1, suggesting they were colored primarily with manganese (Figure 10). The other dark-glass samples from Espace du Palais (not IIa7 beads) contain high amounts of manganese, ranging from 3.4% to more than 13% (Dussubieux 2009:106), and eight purple beads from Cours Napoléon contain high amounts of manganese oxide with concentrations of 4%-11% (Dussubieux and Gratuze 2012:34).

Group 2 beads are the most varied in composition with mixed manganese and cobalt concentrations. Manganese ranges from 3%-9% and cobalt is equally variable, ranging from 150-400 ppm. The glass beads from Asd/kg10, Espace du Palais, and Fort St. Louis all fall within this group. It would be possible to distinguish sub-groups within Group 2, but a larger sample is needed. For the most part these beads have low amounts of arsenic associated with the cobalt.

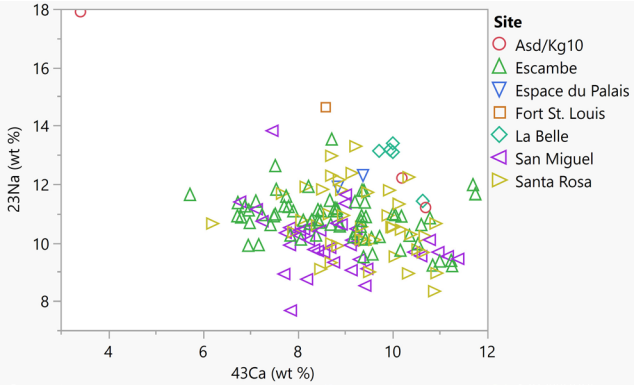

Figure 6. Biplot of soda and lime concentrations in the glass beads (wt. %).

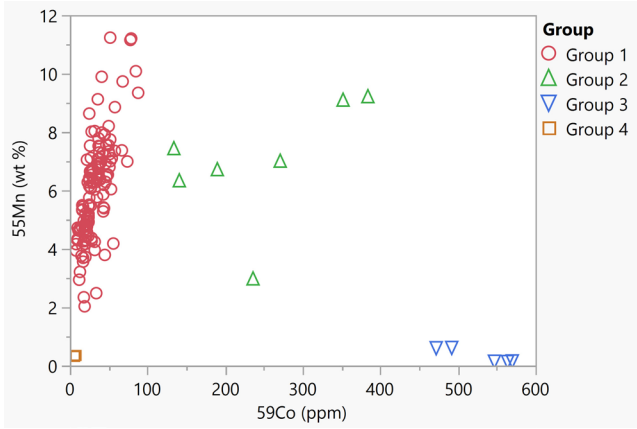

Figure 7. Biplot of manganese and cobalt concentrations in the Pensacola beads (wt. % and ppm).

Unfortunately, there is not enough chemical data to infer the origin of the cobalt ore.

Group 3 beads are colored primarily with cobalt and contain less than 1% manganese. These beads are from Santa Rosa and Escambe, and contain trace amounts of nickel (200-400 ppm), bismuth (200-400 ppm), and arsenic (600-1000 ppm). Studies by Gratuze et al. (1995) were able to identify four compositional groups of cobalt-colored glass based on trace elements, including a cobalt-zinc-lead-indium glass, a cobalt-nickel glass, a cobalt-arsenic-nickel-bismuth glass (smaltite), and a cobalt or cobalt-antimony glass. Smaltite is a cobalt ore found in the Scheeberg-Erzgebirge mining district in Saxony and was used from the 16th to the 18th century, primarily by Bohemian glassmakers (Dussubieux 2009; Gratuze 2013; Gratuze et al. 1995). Smalt was invented in Bohemia and made its way to the Netherlands sometime during the 16th century (Müthlethaler and Thissen 1969).

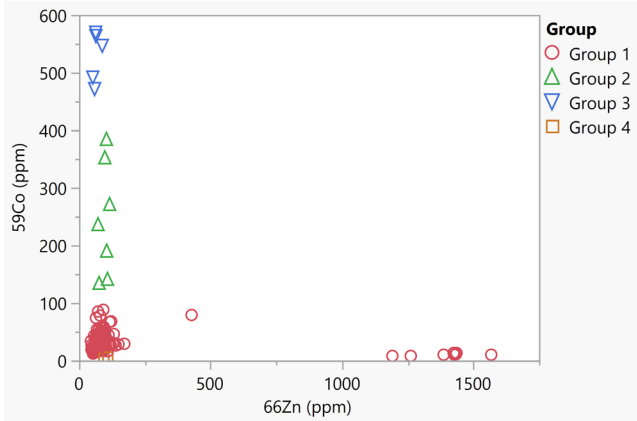

Figure 8. Biplot of cobalt and zinc concentrations in the Pensacola beads (ppm).

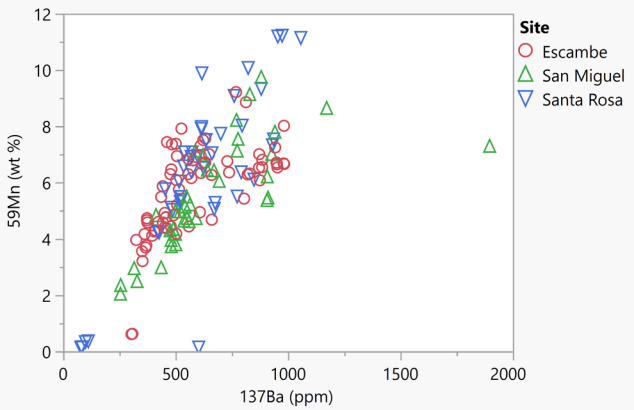

Figure 9. Biplot of manganese and barium concentrations in the Pensacola beads (wt. % and ppm).

Group 4 contains no significant amounts of manganese, barium, or cobalt. It is represented by two beads from Santa Rosa that are the only two beads in the Pensacola assemblage with significant amounts of antimony (see next section for further discussion). These beads also have low iron and copper, making it unclear as to what was used to obtain the black color of the glass.

The Pensacola black beads were partially opacified with trace levels of antimony (Figure 11). The two beads from Group 4 contain 5%-6% with all other groups containing antimony in quantities less than 3500 ppm. Groups 1-3 have trace amounts of tin (0-200 ppm), which correlates with the use of antimony well into the 18th century. The occupations at Pensacola significantly post-date the use of tin as an opacifier. The glass beads from Espace du Palais, Fort St. Louis, and *La Belle* have slightly higher traces of tin, but the Espace du Palais beads are the only ones without any antimony. The beads from the other two sites also contain trace amounts of antimony, suggesting that these beads were opacified during the tin/antimony transitional period.

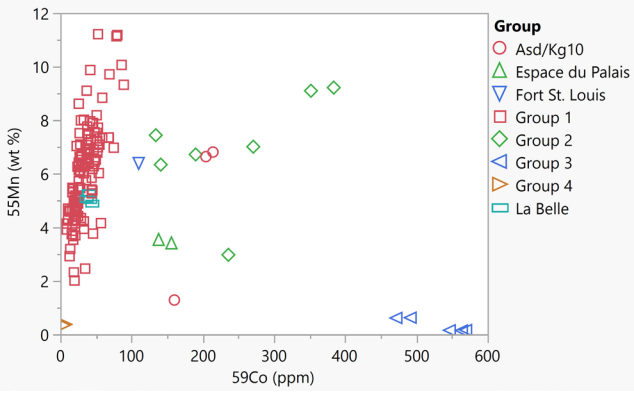

Figure 10. Biplot of manganese and cobalt concentrations in all beads (wt. % and ppm).

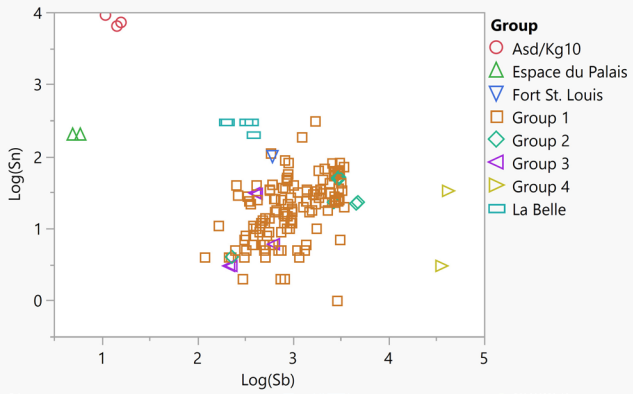

Figure 11. Biplot of tin and antimony concentrations in all beads (log10, ppm).

It could also indicate glass recycling (see discussion below). Furthermore, the beads from Asd/Kg10 have almost no antimony and close to 1% tin. Since this site dates to the 17th century, the data align with previous studies on white beads. The replacement of tin by antimony appears to occur within any opaque bead color, indicating that the switch was likely due to economic pressures rather than ingredient preference (Templin 2017).

Generally, in black glass, traces of other elements – such as lead, tin, copper, and antimony – are present in the glass, but are sometimes unrelated to its color (or opacity). This indicates that the glass was made by remelting or recycling cullet of different colors (Veritá and Zecchin 2008:112). The black beads from Pensacola have trace amounts of lead, copper, tin, and antimony which may indicate recycling. Some samples from France also contain small amounts of the following oxides (up to): copper 0.4%, tin 1.3%, arsenic 0.5%, and lead 1% (Dussubieux and Gratuze 2012:34). This also indicates recycling, which seems to be a common technique for making black glass. Although this analysis is based on small sample sizes and differing analytical strategies, it offers insight into the potential of chemically comparing both synchronic and diachronic datasets.

CONCLUSION

The research presented here represents the starting point for a much larger undertaking. Previous analyses of beads recovered from archaeological sites in North America have focused on the 16th, 17th, and 19th centuries, primarily sampling white, blue, turquoise, and red beads.[2] There is a dearth of information concerning compositional analyses of glass beads from the 18th century, even more so with black glass beads. Chemical analysis of blue and opaque white beads in the Southeast and Great Lakes region has created a foundational dataset with which to compare glass beads

from other archaeological sites, both in North America and Europe. Chronologies based on metal oxides have the potential to be refined using a combination of historical data and an overall larger chemical dataset for comparison (Dadiego, Gelinas, and Schneider 2021). Additionally, since some polychrome glass bead varieties were manufactured and traded for short periods of time, elemental analysis of those beads would allow researchers to sort out similarities and differences in their glass chemistries, contributing significant data to established chronologies (Hancock 2005:52).

Based on this preliminary analysis and comparison, the beads from Pensacola, as well as those from the other sites in the United States, are all soda-lime-silica glass which may be attributed to Venice. The glass beads from Amsterdam and France show a similar composition, and it seems that they were manufactured following similar recipes but acquired raw materials from different locations. A larger sample, as well as data from other glass production centers, is needed to add to the conversation on bead provenience. For the most part, the beads discussed in this study are all colored with manganese, with some beads also containing small amounts of cobalt which could have contributed to their coloring. The preliminary explanation for the presence of trace amounts of tin, antimony, lead, and arsenic is that the black color was also obtained by mixing or recycling different colors of glass cullet.

The chemical analysis from Pensacola, Florida, represents the largest dataset to date of glass seed beads recovered from 18th-century Spanish contexts (Dadiego 2020). Preliminary analysis of the black beads recovered from the Pensacola sites reveals that although they are primarily colored with manganese, the results are more nuanced. These beads deserve a more in-depth analysis to determine the intricacies of coloring, opacifying, and provenience beyond a blanket interpretation of recycled glass. Just as it is possible to determine where the cobalt ore came from based on minor and trace impurities, the same can be done for manganese and manganese-barium ores. Much more chemical analysis of the present collection, as well as beads from contemporary French and English contexts, is needed to fully unravel the complexity of glass bead distribution and consumption in this region. The chemical analysis of the glass bead assemblage from Santa Maria de Galve (Pensacola's first presidio, not reported here) would make an excellent dataset for the comparison of late 17th-century and early 18th-century glass beads from Spanish and non-Spanish contexts discussed in this article. This research has barely scratched the surface and much more work is needed to understand how glass beads in general, and black glass beads in particular, fit into the colonial narrative.

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ENDNOTES

1. Morphological and chemical data are available from the author upon request.

2. *See* Templin's (2017) M.A. thesis for an analysis of over 900 black glass beads from 17th-century mission Santa Catalina de Guale using pXRF.

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