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Glass analysis – J R N Peake and I C Freestone

Methods

A list and brief description of the beads provided for analysis is presented in Table 104 and images are presented in Figures 5.14 to 5.17.

Small samples were taken, mounted in epoxy resin and polished. They were coated with a thin layer of carbon and examined in the scanning electron microscope (CamScan Maxim). The chemical compositions of the samples were determined using an Oxford Instruments INCA energy-dispersive x-ray analyser (EDXA) attached to the SEM. Relative analytical accuracy is believed better than ±2% for silica, and ±5% for other elements present in concentrations greater than 10%, but greater for elements present in lower concentrations. Detection limits were approximately 0.1% for most of the components analysed, 0.3% for lead and tin and 0.4%-0.7% for antimony, depending on the glass matrix. Results were taken from an average of three analyses and were normalised to 100% to improve precision and comparability.

Oxide compositions for the glasses analysed are presented in Tables 105 and 106.

Results

On closer examination of the beads provided for analysis, not all were identified as glass. Bead 7127 is probably bone (Richard Madgwick, pers. comm.) and bead 3673 is amber (Figure 5.14). As only the glass beads are of interest here, these will not be discussed further. Samples could not be obtained from 6261, 6277, 6538 and 7453, so these were not analysed.

Low-lead glass

All of the translucent blue glass beads are soda-lime-silica glasses manufactured using a relatively pure alkali or "natron" source, as indicated by their low potash and magnesia contents. The blue colour of these glasses is likely to derive from small amounts of cobalt. Approximately 0.1% cobalt oxide was detected in beads 4363, 4429 and 4467 (Figure 5.15), but it is likely that in the other blue glasses it is present in levels below the detection limits of EDXA. Only 500 ppm (0.05%) cobalt is sufficient to colour a glass a strong translucent blue (e.g. Freestone *et al.* 2008).

Compositionally all of the blue glasses are very similar, with around 17% Na_2O, 2.5 % Al_2O_3 and 6% CaO, but there are several distinctions within this group. With the exception of 4467, all contain high

amounts of antimony, at around 1.6%. Bead 4467 is slightly different in that it contains no antimony above the detection limits of the technique (c. 0.3%) and has a lower alumina content than the other low-lead blue glasses. It also contains slightly more magnesium, manganese and titanium oxides. This variation in composition suggests that slightly different raw materials were used in its production.

Glass fragment 8117 is unusual (Figure 5.17). It is a soda-lime-silica glass with some compositional similarities to the other low-lead glasses. However, the chlorine content is negligible, which is usually only found in glass manufactured after the introduction of the Leblanc process for the production of synthetic soda in the early nineteenth century. The very high level of manganese is also unusual for an ancient pale green glass. However, other characteristics of this glass are consistent with and even suggestive of glass of the period. At present the affinities of this sample are unclear.

High-lead glass

All of the high-lead glass beads analysed contain similar amounts of lead oxide (75-78% by weight) with approximately 20% silica. This is consistent with previous analyses of contemporary high-lead glass beads, as well as literary works by authors of the time including Theophilus and Heraclius detailing the manufacture high-lead glass (Bayley 1990; Bayley 2009; Henderson and Warren 1986). The low levels of alkali (Na_2O and K_2O) and alkaline earth (MgO and CaO) oxides suggests the use of a simple two-component recipe whereby lead and silica (probably added as sand) were fused together.

The two opaque yellow beads (7344 and 7418; Figure 5.14) are coloured and opacified by crystals of lead-tin oxide homogenously dispersed throughout the glass matrix. Their elemental composition is consistent with the cubic phase $PbSnO_3$ (Rooksby 1964; Tite *et al.* 2008). These crystals probably formed directly in the glass melt from a mixture of silica, lead and tin compounds.

The remaining high-lead glasses are all translucent. Low amounts of iron, copper, manganese and antimony are often found in beads of this type and date, with parallels being found at sites such as Coppergate in York (e.g. Bayley and Doonan 2000). The translucent green glass beads (6837, 7365, 7466 and 8116; Figure 5.14) are all compositionally very similar and contain notable amounts of copper oxide, which is likely to contribute to their colour; copper produces a green colour in high-lead glasses such as these (Bayley 1990). Bead 7733 has a very high iron content (3.4%) which has produced a very dark olive green (appearing black) colour (Figure 5.14). The slightly higher sulphur content in this glass suggests that iron sulphides may be partly responsible for the 'black' colour.

Bead 6833 (Figure 5.16) is particularly unusual because its chlorine and soda contents are both high (1.3%) in comparison to the other high-lead glasses. Given the very low concentrations of other alkali and alkaline earth elements, this strongly suggests the addition of salt (NaCl) to the glass batch.

Discussion

Two broad types of glass have been identified by the present report; one containing low amounts of lead oxide (Table 105) and the other conversely containing high amounts (Table 106). The two glass-working industries are likely to have been contemporary with one another (Bayley and Doonan 2000), but as they share no compositional similarities, it is unlikely that they were directly related. All of the low-lead alkali-silicate glasses analysed (Table 105) are of the low-potash, low-magnesia type manufactured using a relatively pure mineral form of soda ("natron"). The production of raw natron-based glass in the Middle East appears to have ceased in the ninth century (Shortland *et al*. 2006, Phelps et al. 2016). It is therefore likely that these beads represent the re-use of earlier material, rather than the use of fresh glass imported from the Eastern Mediterranean (Freestone 2015); the presence of antimony in most of them indicates a significant content of Roman glass, pre-dating the end of the fourth century when the use of antimony to decolourise and opacify glass had largely ceased (Tite et al 2008). It is possible that this material reached northern Europe in the form of mosaic tesserae stripped from old buildings. Indeed, the compositions of the translucent blue beads closely resemble translucent windows and vessels from sites such as San Vincenzo al Volturno, Italy, where there is clear evidence for a substantial production of glass, based upon the recycling of Roman tesserae and windows (Schibille and Freestone 2013). In northern Europe, the re-melting of tesserae as a raw material in early medieval bead making has been recorded elsewhere, for example at Åhus, Sweden (Callmer and Henderson 1991). Overall, given our expected analytical errors, the glass compositions of 3314, 4363 and 4429 are sufficiently similar to suggest that they may have been products of the same glass batch, 4492 was made using the same approach but from a different batch, while 4467, the square bead, is markedly different.

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Although the translucent glass blob (6277; Figure 5.17) was not analysed, very similar glassworking waste has been found in contexts containing high-lead glass artefacts at Flaxengate in Lincoln, where there is also strong evidence for the re-melting of old Roman glass during the tenth and eleventh centuries (Bayley 2008).

The second class of glass contains high amounts of lead (Table 106). The working of high-lead glass appears to have been introduced to Britain during the tenth century, but becomes comparatively rare in eleventh century contexts (Bayley 2009). Unlike earlier glasses which contain up to 30% lead oxide, this glass type contains approximately 70% (Bayley and Doonan 2000). It was commonly used to make

beads, finger-rings and trinkets, but does not appear to have been used to manufacture vessels until the fourteenth century (Bayley and Doonan 2000).

Evidence for the manufacture of objects using glass of the high-lead type is widespread in the British Isles during this period (Bayley 1990; Bayley 2009). There is an abundance of evidence from sites in Lincoln, York, Gloucester and Dublin from the tenth and eleventh centuries (Bayley 2008; Bayley 2009). Close parallels are also known from Eastern and northern Europe from the ninth century onwards, which is not surprising given the long-distance trade-links established by Viking settlers at the time (Bayley 2008; Bayley 2009; Mecking 2013; Siemianowska et al. 2019). Given that high-lead glass appears to have been comparatively rare in Scandinavia and that it has been commonly found in areas outside of Viking influence in southern England (Bayley 2009), it is likely that the high-lead glass beads were locally manufactured in Britain. However, the possibility that the glass has its origins on the Continent cannot be ruled out.

The vast majority of high-lead glass in Britain is normally translucent yellow or emerald green in colour, but 'opaque' black and, more rarely, blue-green colours are also found (Bayley 2008; Bayley 2009; see also Siemianowska et al. 2019). Small but significant amounts of copper were present in four of the translucent green beads (6837, 7365, 7466 and 8116), which is a typical colourant in this high-lead glass (Bayley and Doonan 2000). It is likely that salt (NaCl) was added in the production of bead 6833, which is mentioned as an ingredient in Theophilus's twelfth century treatise *De Diversis Artibus* in a chapter on the manufacture of high-lead glass finger rings (Hawthorne and Smith 1979). However, it does not appear to have been identified in previous studies of early medieval high-lead glass. Salt also appears to have been commonly added to glass in the later medieval period (e.g. Schalm *et al.* 2007).

The two opaque yellow glass beads (7344 and 7418) are opacified and coloured by crystals of lead-tin oxide, or lead stannate ($PbSnO_3$). The duration of heating would have been minimised as lead-tin yellow is unstable and can readily lose its colour at high temperatures. Opacifiers based on tin were used throughout the early medieval period in the production of yellow glass both in the British Isles and Europe (e.g. Tite *et al.* 2008). In early medieval glass from Anglo-Saxon and Merovingian contexts an opaque yellow glass very similar to the present compositions was added to colour a soda-lime-silica glass (Heck et al. 2003, Peake and Freestone 2014), but this practice was not followed in the present case as the opaque yellow beads have low levels of soda and lime.

Conclusions

All of the glass beads analysed are compositionally consistent with contemporary early medieval glass used in Britain from the tenth to twelfth centuries AD, with the exception of glass fragment 8117 which

has some unusual features. Two types of glass have been identified from the present assemblage; a high-lead glass and a soda-lime silica glass. All of the translucent blue glasses fall into the latter category and appear to represent the re-use of opaque blue Roman glass, probably tesserae.

The remaining glass beads, which include translucent green, translucent yellow, opaque yellow and 'black' colours, contain high levels of lead oxide. Their composition is paralleled by previous analyses of high-lead glass beads from elsewhere in the British Isles (e.g. Lincoln) and in tenth to twelfth century glass from elsewhere in northern and central Europe. The low alkali contents suggests that they were manufactured using a two-component system, whereby lead and silica were fused together and a colourant was added if desired. The green beads are coloured by copper and the 'black' bead by iron. The opaque yellow beads are both coloured and opacified by lead-tin oxide, which is typical of opaque yellow glass produced after the fourth century AD, prior to which opaque yellow glass was commonly based on compounds of antimony. The translucent yellow glass bead contains higher levels of both soda and chlorine than the other high-lead glasses. This suggests the use of salt (NaCl) in its production; a practice which is mentioned by Theophilus in his twelfth century treatise on the manufacture of high-lead glass.

The soda-lime-silica beads were made from old Roman glass which was probably imported from southern Europe, possibly as tesserae. The glass of the lead-silica beads is of a type widespread in Europe, but it is possible that it was made in the region, or even locally. More detailed compositional studies of lead-silica glass of the period would be required to determine any regionally distinctive groupings and their possible origins.

References

Bayley, J. (1982) 'Non-Ferrous Metal and Glass Working in Anglo-Saxon Scandinavian England: An Interim Statement', PACT, **7**(2), 487-96.

Bayley, J. (1987) 'Viking Glassworking: The Evidence from York', *Annales du 10e Congrès de l'Association Internationale pour l'Histoire du Verre*, 245-254.

Bayley, J. (1990) 'Glass Rings and the Manufacture of High-Lead Glass', in Biddle, M. (*ed.*) *Object and Economy in Medieval Winchester*, 268-269. Oxford: OUP (Winchester Studies 7.ii).

Bayley, J. (2008) *Lincoln: Evidence for Glass-Working on Flaxengate and Other Sites in the City* (Research Department Report 68/2008). Portsmouth: English Heritage.

Bayley (2009) 'Early Medieval Lead-Rich Glass in the British Isles – A Survey of the Evidence', *Annales du 17e Congrès de l'Association Internationale pour l'Histoire du Verre*, 255-260.

Bayley, J. and Doonan, R. (2000) 'Glass Manufacturing Evidence', in Mainman, A. and Rogers, N. *Craft, Industry and Everyday Life: Finds from Anglo-Scandinavian York*, 2519-2528. York: CBA (Archaeology of York 17/14)

Callmer, J. and Henderson, J., (1991) 'Glassworking at Åhus, S. Sweden (eighth century AD)', *Laborativ arkeologi*, **5**, 143-154.

Freestone, I.C., (2015) 'The recycling and reuse of Roman glass: analytical approaches', *Journal of Glass Studies*, **57**, 29-40.

Freestone, I., Hughes, M. and Stapleton, C. (2008) 'The Composition and Production of Anglo-Saxon Glass', in Evison, V. and Marzinzik, S. (*eds.*) *Catalogue of Anglo-Saxon Glass in the British Museum*, 29-46. London: The British Museum.

Heck, M., Rehren, T. and Hoffmann, P. (2003) 'The Production of Lead-Tin Yellow at Merovingian Schleitheim (Switzerland)', *Archaeometry*, **45**(1), 33-44.

Henderson, J. and Warren, S. (1986) 'Analysis of the Glass and Glassy Waste', in Tweddle, D. *Finds from Parliament Street and Other Sites in the City Centre*, 224-226. York: CBA (Archaeology of York 17/4).

Mecking, O. (2013) 'Medieval lead glass in central Europe', *Archaeometry*, **55**(4), 640-662.

Peake, J.R. and Freestone, I.C. (2014) 'Opaque yellow glass production in the early medieval period: new evidence', *Neighbours and successors of Rome: traditions of glass production and use in Europe and the Middle East in the later 1st millennium AD*, pp.15-21. Oxford: Oxbow Books.

Phelps, M., Freestone, I.C., Gorin-Rosen, Y. and Gratuze, B. (2016) 'Natron glass production and supply in the late antique and early medieval Near East: The effect of the Byzantine-Islamic transition', *Journal of Archaeological Science*, **75**, 57-71.

Rooksby, H. (1964) 'A Yellow Cubic Lead Tin Oxide Opacifier in Ancient Glasses', *Physics and Chemistry of Glasses*, **5**, 20–5.

Schibille, N. and Freestone, I.C. (2013) 'Composition, production and procurement of glass at San Vincenzo al Volturno: an early medieval monastic complex in Southern Italy', *PloS one*, **8**(10), e76479.

Siemianowska, S., Pankiewicz, A. and Sadowski, K. (2019) 'On technology and production techniques of early medieval glass rings from Silesia' *Archaeometry*, **61**(3), 614-646.

Tite, M., Pradell, T. and Shortland, A. (2008) 'Discovery, Production and Use of Tin-Based Opacifiers in Glasses, Enamels and Glazes from the Late Iron Age Onwards: A Reassessment', *Archaeometry*, **50**(1), 67-84.

Table 104– Descriptions of the Bornais beads

SF Number	*Mound*	*Context*	*Material*	*Description*	*Glass colour*[1]
3314	2	1048	Glass	Bead	tr. blue
3673	2	1048	Amber	Bead	-
4363	2	1257	Glass	Bead	tr. blue
4429	2	580	Glass	Bead	tr. blue
4467	2	1051	Glass	Bead	tr. blue
4492	2	581	Glass	Bead	tr. blue
6261	2A	2397	Glass	Bead	op. yellow
6277	2A	2413	Glass	Nugget/blob	tr. 'colourless'
6538	2	2668	Glass	Bead	tr. blue
6833	2A	2441	Glass	Bead	tr. yellow
6837	2	599	Glass	Bead	tr. green
7127	2A	2316	Bone	Bead	-
7344	2	2210	Glass	Bead	op. yellow
7365	2	2281	Glass	Bead	tr. green
7418	2	2251	Glass	Bead	op. yellow
7453	2	2042	Glass	Bead	tr. green
7466	2	2697	Glass	Bead	tr. green
7733	2	2669	Glass	Bead	'black'
8116	2	2281	Glass	Bead	tr. green
8117	-	20	Glass	Vessel fragment?	tr. p. green 'colourless'

[1]tr. = translucent; op. = opaque; p. = pale.

Table 105 – SEM-EDS analyses of the low-lead glasses

Oxide (wt%)[1]	*Sample*[2]					
	3314 *tl. blue*	***4363*** *tl. blue*	***4429*** *tl. blue*	***4467*** *tl. blue*	***4492*** *tl. blue*	***8117*** *tl. p. green*
Na_2O	17.2	17.0	16.8	18.3	18.9	15.6
MgO	0.7	0.6	0.7	1.1	0.5	0.4
Al_2O_3	2.2	2.2	2.3	1.7	2.0	2.4
SiO_2	66.4	66.9	65.9	68.2	67.5	66.0
P_2O_5	0.2	0.2	0.2	0.2	b.d.	b.d.
SO_3	0.5	0.4	0.3	0.2	0.4	0.5
Cl	0.9	0.9	1.0	1.3	1.0	0.1
K_2O	0.9	0.6	0.8	0.9	0.6	0.4
CaO	6.4	6.5	6.7	6.2	5.6	8.2
TiO_2	0.1	0.1	0.1	0.2	b.d.	0.3
MnO	0.5	0.5	0.5	0.8	0.2	3.9
Fe_2O_3	1.1	1.1	1.2	1.1	0.9	1.7
CoO	b.d.	0.1	0.1	0.1	b.d.	b.d.
CuO	0.8	tr.	0.8	tr.	tr.	tr.
Sb_2O_3	1.7	1.7	1.6	b.d.	1.5	b.d.
PbO	0.4	0.4	0.6	b.d.	b.d.	b.d.

[1]b.d. = below detection. tr. = trace. Detection limits were thought to be about 0.1% for most of the elements analysed, although this is marginally higher for lead and tin at about 0.25-0.3% and rises to over 0.5% for antimony in glasses with high calcium. Zinc oxide and tin oxide were analysed for but not detected.

[2]tl. = translucent; p. = pale.

Table 106 – SEM-EDS analyses of the high-lead glasses

Oxide (wt%)[1]	*Sample*[2]							
	6833 *tl. yellow*	***6837*** *tl. green*	***7344*** *op. yellow*	***7365*** *tl. green*	***7418*** *op. yellow*	***7466*** *tl. green*	***7733*** *'black'*	***8116*** *tl. green*
Na_2O	1.3	b.d.	b.d.	b.d.	0.2	b.d.	b.d.	0.2
MgO	b.d.	0.1	0.2	b.d.	b.d.	0.2	b.d.	0.2
Al_2O_3	0.4	0.2	0.6	0.3	0.5	0.5	0.5	0.6
SiO_2	21.0	21.6	17.5	22.0	18.2	22.3	20.0	20.0
P_2O_5	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	0.2	b.d.
SO_3	0.2	0.1	0.1	b.d.	0.2	b.d.	0.4	b.d.
Cl	1.3	0.1	b.d.	b.d.	b.d.	0.2	b.d.	0.1
K_2O	b.d.	b.d.	0.2	b.d.	0.2	0.2	b.d.	0.1
CaO	b.d.	b.d.	0.2	0.2	0.2	b.d.	0.2	0.5
TiO_2	b.d.	0.2	b.d.	b.d.	b.d.	b.d.	0.1	b.d.
MnO	b.d.	b.d.	b.d.	b.d.	0.1	b.d.	0.2	b.d.
Fe_2O_3	0.2	b.d.	0.3	0.3	0.2	b.d.	3.4	0.2
CuO	tr.	1.2	tr.	0.8	tr.	1.4	b.d.	1.1
ZnO	b.d.	0.1	b.d.	0.2	0.3	0.2	0.2	b.d.
SnO_2	b.d.	b.d.	3.3	b.d.	1.6	b.d.	b.d.	b.d.
PbO	75.5	76.6	78.4	76.4	78.0	75.8	75.2	77.3

[1]b.d. = below detection. tr. = trace. Detection limits were thought to be about 0.1% for most of the elements analysed, although this is marginally higher for lead and tin at about 0.25-0.3% and rises to over 0.5% for antimony in glasses with high calcium. Cobalt oxide and antimony oxide were analysed for but not detected.

[2]tl. = translucent; op. = opaque.