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ANALYSIS AND INTERPRETATION OF GLASS ARTEFACTS FROM 17TH– 19TH CENTURY DUTCH AND COLONIAL AUSTRALIAN SHIPWRECKS

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ABSTRACT

Australia's historic shipwrecks, which date from 1622 to 1923, provide a rich source of glass bottles and other glass artefacts. Semi quantitative and quantitative analysis of the glass has shown up some systematic differences in the composition from the early seventeenth century until the nineteenth century. The limited amount of data from some of the early Dutch shipwrecks indicates the use of two principal types of potash glass while the pre-colonial and early post-colonial period shows up a different range of compositions. Glass from wrecks dating from the 1850's are generally dominated by soda-lime compositions. Quantitative analysis of glass from the 1790 wreck of HMS *Sirius* on Norfolk Island shows how the chemical composition and refractive index can provide a very handy archaeological classification tool. Analysis of the distribution of impurities has provided a guide to the origin of the raw materials used in the manufacture of the glasses.

KEYWORDS

Glass composition, refractive index, leaded crystal, window glass, shipwrecks, bottles

INTRODUCTION

Over the past twenty five years a series of shipwreck sites have been investigated and partly excavated by the teams of maritime archaeologists and conservators from the Western Australian Museum in Fremantle. Despite several studies by conservators on the issues relating to desalination and consolidation [1–4] scant attention has been paid to the analysis and interpretation of the glass fragments which are the common form of glass artefacts recovered from high energy wreck sites. While analysis of the contents of bottles and jars has revealed a variety of ciders, wines and pickles [5,6] which has seen an increased understanding of the role of commerce in consumables, the number of studies on the analysis of the glass vessels and drinking glasses is very limited Despite detailed archaeological documentation and descriptions there has been remarkably little chemical analysis done on the fragments of bottles and panes which are eminently suitable for sampling since they are already damaged. The erosion patterns can also provide an insight into the coastal geomorphological changes. The 5 mm banded layers of devitrified glass from the *Zuytdorp* (1712) wreck reflect the changes in the physical environment as massive amounts of sand are regularly moved by storms. This action alternatively buries and exposes parts of the wreck [4]. Similarly corrosion layers on a brass fitting from the wreck of the SS *Xantho* (1872), some 100 nautical miles to the south, show that the cyclic burial and exposure tends to follow a seven year pattern [7]. In order to address the dearth of data on our glass heritage a selection of seventeen bottle and jar fragments were subjected to Scanning Electron Microscopic (SEM) examination prior to the availability of quantitation software. Since the semi-quantitative analysis from the Energy Dispersive X-Ray Analysis (EDXRA) provided a useful set of data, later studies on twenty samples of glass from the wreck of HMS *Sirius* (1790), the flagship of the First Fleet of white Australian settlers, were performed in a quantitative fashion. The wreck of the *Sirius* is located at Norfolk Island in the South Pacific Ocean.

The glass recovered from the Western Australian shipwrecks is inherently interesting because it covers a period of manufacture from the early seventeenth century with materials from the *Batavia* (1629) and *Vergulde Draeck* (1656) to the eighteenth century which is represented by materials from the *Zuytdorp* (1712) and the *Zeewijk* (1727) wreck as well as materials from the *Sirius*. Glass from the wreck of the American China Trader *Rapid* (1811) fills the gap between the end of the Dutch Eastindiamen and the vessels of the colonial period which started in 1829. The *Cumberland* (1830), *James Matthews* (1841), *Xantho* (1872) and the *Carlisle Castle* (1899) complete the range of vessels of the colonial period. Four samples of glass bottles recovered from the site of the long jetty, which serviced ships in the Fremantle area from 1870–1897 complete the range of materials which were examined. Thus the period of the glass artefacts spans a time frame during which major changes in industrial practices in England and in the European continent took place.

Analysis of glass

Core samples were prepared for analysis by careful removal of any exfoliant and highly degraded layers. Quantitative data reported in the tables as weight percent oxide compositions are the average of triplicate measurements. Core samples of the fragments from the

Sirius gave totals of between 99.9 and 99.0%, with the most common value being 99.45±0.22 wt%. Quantitative oxide concentrations were calculated by stoichiometry from element percentages generated by ZAF-4FLS software on the Oxford-Link eXL EDXRA.

Semi-quantitative analysis of shipwreck glass artefacts

Analytical data from the seventeen samples of glass from Western Australian wrecks is tabulated according to the relative abundance of the elements in the categories of major, minor and trace amounts. The data is listed chronologically in terms of the actual date of the shipwreck since this provides a handy guide to the age of manufacture of the glassware. It is likely that the glass artefacts recovered from the shipwrecks date from the time the ship was loaded since the description generally shows the objects were a part of the cargo.

In the case of the *James Matthews* (JM 313) the window glass is not from the vessel itself but it formed part of the cargo manifest of goods being brought to Western Australia for the infant colony. For the wreck of the SS *Xantho*, the thick plate glass may have come from the engine room fittings and could therefore have pre-dated the wreck by twenty years. Inspection of the elemental distributions shown in Table I shows that most of the glass objects were lime based glass. The presence of titanium in trace amounts in twelve glasses may be an indicator of the use of beach sands as the source of silica for those samples since the black mineral sand ilmenite, $FeTiO_2$, is commonly distributed in marine based sands.

Despite the absence of a quantitation package, the X-ray counts were used to provide a guide to the composition of the glasses by manually recording the intensity of the elements on the printouts and comparing the ratios of the silicon to calcium and potassium peaks. The count time was adjusted to ensure that the same number of counts for silicon was recorded. Similar ratio analysis provided a limited set of ratios for silica and sodium and magnesium but since these elements were not always present at a "minor" level, it is difficult to see if there are any systematic trends. Despite the limited data base, the distribution of the ratios showed a general trend that the magnesium and sodium values were mimicking each other.

Of greater significance is the grouping of the ratios of $^{Ca}/_{Mg}$ which clustered around a value of 24.6±3.5 for all the Dutch glass from the *Batavia* (BAT), *Vergulde Draeck* (GT), *Zeewijk* (ZW), *Zuytdorp* (ZT) and for the glass from the American ship *Rapid* (RP). The ratios for wrecks after 1811 had either very much higher or lower values. It is

Table 1 Qualitative elemental analysis of Western Australian shipwreck glass

Regn no.	*Date*		*Major*	*Minor*	*Trace*
BAT 4620	1629	dark glass	Si,K,Ca	Na,Al,Mn,Fe	P,S,Ti
GT 4006	1656	green case gin	Si,K,Ca	Mg,Al,Mn	P,S,Ti
ZT 4054	1712	dark bottle	Si, Ca	Na,Mg,Al,Cl,K,Fe	P,S,Ti,Mn
ZW 1873	1727	green onion	Si, Ca	Mg,Al,Cl,K	Na,P,Ti,Mn,Fe
ZW 2063	"	mauve tumbler	Si,K,Ca	Mg	S,Cl,Ti,Mn
ZW 3000	"	green case gin	Si, Ca	Na,Mg,Al,K,Fe	P,S,Ti,Mn
ZW 3102	"	wine glass stem	Si, Pb,K	-	Cl
RP 3011	1811	dark bottle	Si, Ca	Mg,Al,Cl,K,Fe	Na,S,Ti
CM 12	1830	dark bottle	Si, Ca	Mg,Al,K,Fe	Na,S,Cl,Ti
JM 313	1841	window	Si, Ca	Na	Al,S,Cl,K,Mn
EG 1504	1852	dark bottle	Si, Ca	Mg,Al,K,Fe	Na,S,Cl,Ti
XA 209	1872	window	Si, Ca	Na	S,Cl,K
LJ 070	1870-1897	green	Si, Ca	Na,Mg,Al,K,Fe	S
LJ 134	"	green	Si, Ca	Na,Mg,Al,Cl,Mn,Fe	S,K,Ti
LJ 488	"	green	Al,Si,K,Ca	Na,Fe	S,Cl,Ti,Mn
LJ 613	"	green	Si, Ca	Na,Al,Mn,Fe	Mg,S,Cl,K,Ti
CA 3167	1899	clear pickle	Si, Ca	Na	S,Cl,K,Fe

likely that the common ratio of the calcium and magnesium for these early shipwreck glasses indicates that dolomite $CaMg(CO_3)_2$ was the source of the alkaline earth elements [8]. Apart from the exception of the *Zeewijk* mauve glass ZW 2063 and the leaded crystal glass ZW 3102, all the Dutch and the American glass bottles had a $^{Si}/_{Ca}$ ratio of 2.06±0.17 which indicates that they were using similar recipes for bottle manufacture from the seventeenth until the early nineteenth centuries. Within the scope of the generally similar composition of the glasses, cluster analysis of the $^{Si}/_{K}$ ratios shows up three major groupings for the Dutch and the American vessels. The *Batavia, Vergulde Draeck* samples and the *Zeewijk* samples ZW 2063, 3102 had a mean $^{Si}/_{K}$ ratio of 4.0±0.9 while the other three Dutch samples ZT 4054 and ZW 1873, 3102 had a $^{Si}/_{K}$ ratio of 18.0±1.0. This indicates that the Dutch glass from the seventeenth until the early eighteenth century was based on two traditional potash recipes. The glass from the *Rapid* (1811), *Cumberland* (1830) and the *Eglinton* (1852) had a mean $^{Si}/_{K}$ ratio of 32.1±3.3. The Long Jetty green bottle LJ 70 had a $^{Si}/_{K}$ ratio of 48 while the remaining glasses had ratios from 100-290. The latter results indicate that the bottles LJ 134, LJ 613, the window glass JM 313 and the *Carlisle Castle* (CA) 3167 could all be classified as typical soda glasses where Na_2CO_3 has been used to flux the sand and the lime. There was only one bottle which had a significantly different composition and that was the green bottle from the Long Jetty LJ 488 which had a relatively large amount of aluminium present. The only glass with any significant amount of arsenic was the leaded wine glass stem ZW 3102.

Because of the relative mobility of the sodium in shipwreck glasses, the soda content tends to be underestimated if the surface materials examined have been significantly affected by hundreds of years of immersion in sea water. There are also small but significant amounts of manganese oxides in the glass but this appears to be present largely as a result of the manufacturing processes rather than from anthropogenic sources such as bacteria [4]. In order to see if the presence of iron was largely responsible for the green colour of seven of the samples, the ratios of $^{Si}/_{Fe}$ was determined along with the ratio of counts for $^{Si}/_{Mn}$ since manganese has can exert a major influence on the oxidation state and colour of the iron impurities [9]. The mean value of the $^{Si}/_{Fe}$ ratio was 106±38 which was the same as the mean value of 100±39 for the ratio of the counts of silicon and manganese. The dark brownish coloured glass from the *Batavia, Zuytdorp*, *Rapid*, *Cumberland* and *Eglinton* had lower $^{Si}/_{Fe}$ ratios of 65±16 which is consistent with the deeper colour being due to more iron. Inspection of the data shown in Table I also shows up the presence of trace amounts of sulphur in several samples. The effect of sulphur and iron impurities on the colour of glasses has shown that the presence of sulphur, either from pyrites FeS_2 or from reduced sulphates has a major influence on the colour of the glass [10]. The average ratio of $^{Fe}/_{S}$ for the green glass bottles was 4.7±2.4 while the mean value for the dark glass was not statistically different at 3.2±1.9 but it should be noted that the dark glass bottles had much lower amounts of manganese present and this clearly has a major impact on the colour that the combined presence of iron and sulphur is able to effect.

Quantitative analysis of glass from the wreck of HMS *Sirius* (1790)

A total of twenty samples of glass, which represented the total of 35 different sets of registered fragments of glass [11], were analysed after carefully sampling the glass to create triplicate samples of the broken fragments. The materials were selected from amongst all the shards with the majority of pieces apparently coming from sheets of glass, storage vessels and drinking glasses from the officers' quarters. In order to present the results in a manageable format, the data has been grouped into categories of lead free and low leaded glasses in Table II while Table III is for the lead "crystal" which was found on the site.

The samples SI 217-1 and 217-2 were green glass bottle fragments. The green colour is most likely associated with iron contents of 1.6% and 1.4%, present as FeO, and despite extensive desalination the glass retained 0.7% chloride as measured in the form of Cl_2O. Minor constituents of the bottle glass were approximately 0.35% TiO_2 with the first sample having 2.8% phosphorus and the second sample 1.9% phosphorus as P_2O_5. The high level of phosphorus would impart a greater stability to the glass which would help it to resist the worst of the ravages of 200 years of exposure to constant wave action since the *Sirius* site lies on top of a reef platform in 1.5-4.5 metres of water. The high calcium content of the bottles make them typical of cheap domestic ware of the late nineteenth century. There are sufficient differences in the chemical composition of these two bottle fragments to be certain that they have come from different bottles, even though they were found in close proximity to each other on the wrecksite.

The three samples noted 155-2 (3.7 mm thick clear glass sheet), 155-3 (4.2 mm thick clear glass, surface abraded) and 182-1 (somewhat eroded clear glass 3.5 mm thick) clearly belong to the same category of composition with the same physical form of glass sheet and the same overall chemical composition. The tightness of the values of the refractive index for 155-2 and 182-1 and the identical chemical balance, within experimental error, indicates that they are from the same pane of glass or at least from the same batch. This is further attested by having the same thickness. The thicker sample 155-3 has a slightly higher refractive index and a higher potash content and almost 2% higher lime content - this would appear to be consistent with coming from the same factory but a different batch since the lead contents were very close and this would have been the more carefully measured out component of the composition.

The higher lead content of SI 182-2 compared with the other window glasses SI 182-1 and 155-2 has not affected the refractive index since all three samples have the same value of 1.539±0.001. It is possible that the 3.7% higher lead content of SI 182-2 was unintentional - the lower lime content of 5.3% compared with 7.8% is also probably due to variations in between batches of glass from the same factory. Thus from the four samples of window glass analysed from the wreck site, there are only two thicknesses and one basic chemical composition with some significant degree of variation from batch to batch. The glasses had average chlorine content of 0.80±0.08% present as Cl_2O.

Table II Analysis of lead free and medium leaded glass from HMS *Sirius* (1790)

Regn no.	*RI*	*Na_2O*	*K_2O*	*MgO*	*MnO*	*CaO*	*SiO_2*	*PbO*
SI 217-1	1.576	2.8	2.8	2.8	0.7	25.8	54.5	0.0
SI 217-2	1.576	2.8	2.1	3.8	1.4	24.8	56.9	0.0
SI 155-2	1.538	7.5	9.5	2.0	0.6	7.8	61.0	9.9
SI 155-3	1.544	6.7	10.6	2.1	0.7	9.8	58.6	9.9
SI 182-1	1.539	6.6	9.7	1.9	0.8	8.1	60.7	10.4
SI 182-2	1.539	5.4	10.7	1.2	0.7	5.3	61.4	13.6
SI 290.5	1.553	5.0	12.4	1.3	0.0	6.4	56.7	16.4
SI 155-1	1.566	5.4	11.8	1.7	1.2	5.0	51.8	22.1

Despite the higher refractive index of SI 290-5 due to the higher lead content it is possible that the differences in composition may be little more than poor quality control, if the glass came from the same sources as the other sections of glass which had similar amounts of soda and magnesia. It is possible that the 2.5 mm thick sample of glass (SI 290-5) was prepared according to the same basic formula as the other leaded potash and soda glasses since it has a similar composition as far as the content of the alkali and alkaline earths are concerned. The thinner section glass may have come from a decanter or some such similar artefact. All the four glass samples (155-2, 155-3, 182-1 and 182-2) have a similar $^{Ca}/_{Mg}$ ratio of 4.4±0.4 which is statistically significantly different to SI 155-1 which has a ratio of 2.9. This indicates that a different source of calcium was used in the manufacture of this glass. The glass sample SI 155-1 was badly eroded and was 2.8 mm thick - the higher lead content naturally produces a higher refractive index. Given the apparent closeness of the control over composition of the previous group of leaded glasses, as indicated between the four samples of low leaded glass sheet with PbO contents of 11.0±1.8 wt%, it is most unlikely that the SI 290-5 sample with 16.4% PbO represents an entirely different batch and style of glass. If we examine the ratios of potash over soda, the first three leaded glasses have the ratio of 1.46±0.15 while the SI 182-2, 290-5 and 155-1 have a ratio of 2.23±0.15 which indicates that there are two different sources of alkali being used in the manufacture of these five glasses.

Inspection of the data shown in Table III for twelve high leaded samples of glass artefacts recovered from the wreck of the *Sirius* have a mean lead content of 36.55±0.99 wt % as PbO. The samples all apparently fit into a general category of high quality wine glasses from the officers' quarters while the window glass (listed in Table II) would have come from the great cabin in the stern of the vessel.

A more detailed examination of the data indicates that there are at least two main subgroups based on the amount of potash, with secondary classification being the amount of lead. One group has a mean potash content of 10.12±0.71% while the other group had a mean value of 8.83±0.20%: the latter group includes the materials SI 290-3, SI 385-1&2. The classification is based on the fact that the differences in the mean potash content is more than twice the sum of the standard deviations of the mean. There is less discrimination in the lead content of the these two groups of artefacts with values of 36.14±0.71% and 37.77±0.59% PbO for the group of three glass fragments. The glass fragment SI 184-1 was characterised by the presence of a very fine, 0.15 mm width, engraved feather pattern design which was filled with a lemon-yellow lead mineral, which could not be identified. The design was studied by a glass engraver who determined that this *Sirius* glass has been engraved using a traditional copper disc [14].

The glass fragments SI 290-3, 385-1 and 385-3 were all classified as being heavily eroded by the surge and the surf of the wreck site. In terms of the maritime archaeological interpretation of the data, it is important to be able to discriminate between the different types of materials present in the confused pattern of glass fragments which are readily scattered across the wreck site. Based on the chemical compositions listed in Table III there were clearly two types of heavy leaded "crystal" being carried on the vessel. Since quantitative chemical analysis, even using SEM/EDXRA, is costly and the sample preparation involves breaking a tiny fragment off to mount it on a stub for SEM analysis, it is useful to see if there is a less invasive

Table III Analysis of high leaded glass from HMS *Sirius* (1790)

Regn no.	*RI*	*Thickness mm*		Cl_2O	K_2O	*CaO*	SiO_2	*PbO*
SI 155-4	1.576	16-5.3	curved	0.4	9.3	0.2	53.1	36.3
SI 155-5	1.573	3.2	curved	0.3	10.4	0.0	52.7	35.2
SI 155-6	1.570	2.7	curved	0.4	10.3	0.3	52.3	36.0
SI 184-1	1.574	2.7	curved	0.0	10.3	0.0	52.4	36.4
SI 201-1	1.584			0.4	9.9	0.5	50.9	36.6
SI 290-1	1.574	2.3	flat	0.4	10.2	0.0	52.4	36.3
SI 290-2	1.575	1.7	flat	0.4	10.2	0.4	52.2	35.8
SI 290-4	1.580	3.3	curved	0.4	10.0	0.4	51.1	37.5
SI 385-2	1.575	3.4	wine glass	0.0	10.5	0.0	53.3	35.2
SI 290-3	1.580	4.4		0.6	8.6	0.0	51.1	38.2
SI 385-1	1.579		glass base	0.0	8.9	0.0	52.2	38.0
SI 385-3	1.578	6.5	curved	0.0	9.0	0.0	52.6	37.1

method to assess the type and composition of the glass. The refractive indices of the leaded glasses listed in Tables II & III were plotted as a function of the weight percent lead oxide and the plot can be seen in Figure 1 which shows the strong correlation between the two parameters.

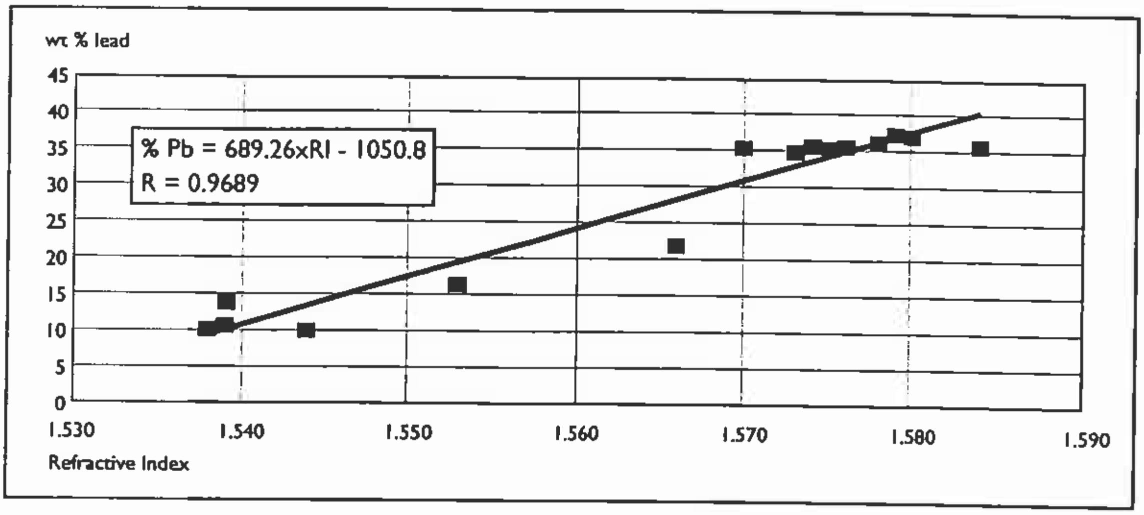

Figure 1
Effect of percentage PbO on the refractive Index on glass samples from HMS *Sirius*.

Although not a perfect fit (as shown by the correlation coefficient being 0.9689) the eighteen data points show that the weight of lead in the glass is related to the refractive index by the relationship,

$$\boxed{\%\ \text{Lead (all glass)} = 689.26 \times \text{RI} - 1050.8}$$

where RI is the refractive index of the glass. When the data is grouped according to the presence or absence of soda, the equation for the leaded glasses which contain both Na_2O and K_2O (mean alkali content 16.9±0.6 wt%) has a lower correlation coefficient of 0.9256 and the equation is given by

$$\boxed{\%\ \text{Lead (soda/potash)} = 405.3 \times \text{RI} - 613.1}$$

which is essentially identical to the literature data [12] for a 25% K_2O glass series where the effect of the weight of PbO on refractive index gives the equation

$$\boxed{\%\ \text{Lead (Newton)} = 406.2 \times \text{RI} - 606.2}$$

An inspection of the residuals plot shows that the greatest cause of deviation from the expected response was found for samples SI 201-1 which had a brown tinge to the glass. Detailed SEM examination of this sample of brown glass showed that it had trace amounts of copper, iron, calcium as well as indications of manganese, cobalt and nickel. It is quite likely that the cocktail of these trace impurities is responsible for the particular colouring of the glass. For the sample SI 290-5 which had the highest potash content of 12.4% there was also a significant deviation from the expected value of lead based on the refractive index. Despite these shortcomings the above relationship (equation 1) may prove to be a useful guide in determining the nature of future finds when the final stages of the site excavation program are in hand. The addition of equipment for measuring refractive index is clearly an important step forward in minimising confusion with regard to proper identification of glass fragments on shipwreck sites.

CONCLUSION

Despite the lack of a quantitation package for the early SEM examination of glasses recovered from Western Australian shipwreck sites, it was possible to determine some systematic trends in composition according to the date of the shipwreck. The presence of trace amounts of titanium indicated a beach sand source for glass from a number of different wrecks. From a comparison of the ratios of the X-ray counts for potassium and silicon it was possible to discern that two formulae of glass manufacture were being used for potash glasses from the 17th to the early 18th century. Early American and colonial period glass tended to have a similar composition which gave way to "modern" soda-lime glasses of the second half of the 19th century. The colour of the glasses is dominated by the amount of iron present with green glass being associated with higher proportions of sulphur and manganese while the brown glass had noticeably different amounts of these modifying agents present. A combination of quantitative elemental analysis of glass from the wreck of HMS *Sirius* has shown that chemical analysis and the refractive index are a very powerful tool in determining the nature of the glass and to show that several different sources of manufacture or batch processes have been involved in the production of the materials recovered on the wrecksite. The direct linear relationship between refractive index and lead content of the glasses can be used as a guide to a better classification of objects.

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