The spectrometric analysis of Iron Age glass beads from Novo Mesto, Slovenia

Spektrometrične analize železnodobnih steklenih jagod iz Novega mesta, Slovenija

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Abstract
This paper presents the results of spectrometric analysis of Iron Age glass from Novo Mesto, Lower Carniola, Slovenia. Several different glass types were detected in the assemblage. The results indicate that raw glass was imported to Novo Mesto from eastern Mediterranean centres and corroborate the existence of long-distance trade during the first millennium BCE.
Key words: glass, chemical composition, provenance, Iron Age, Novo Mesto

Izvleček
Članek predstavlja rezultate spektrometričnih analiz stekla z najdišč starejše železne dobe v Novem mestu, Dolenjska, Slovenija. V izbrani skupini je bilo mogoče opredeliti več različnih tipov stekla. Rezultati kažejo, da je bilo surovo steklo v Novo mesto uvoženo iz središč vzhodnega Sredozemlja in dokazuje obstoj trgovine na dolge razdalje v času 1. tisočletja pr. n. št.
Ključne besede: steklo, kemijska sestava, izvor, železna doba, Novo mesto

Introduction

With its abundant glass bead assemblage, Novo Mesto is one of the key sites for prehistoric glass studies in Europe. No remains of glass production workshops have yet been archaeologically confirmed. However, the myriad of glass items, colour and bead type combinations, attested sand sources suitable for glassworking, and documented knowledge of pyrotechnology during the Early Iron Age (cf. Gabrovec 1987, 93, 95; Haevernick 1974, 65; Križ 2009, 103; Križ and Guštin 2015, 49-50) have suggested that local glassmaking workshops existed in Novo Mesto.

In this short paper, we present the first quantitative trace element data on the Novo Mesto glass assemblage that offers a fresh perspective on the region's prehistoric glass use and production. The project set out to define the compositional types in Novo Mesto and test the local provenance hypothesis through archaeometry.

Analysis

The analysed material consists of forty-eight Late Bronze Age to Late Iron Age (ninth– to second–century BCE) glass beads and one Late Iron Age glass bracelet from the Kapiteljska Njiva and Mestne Njive cemeteries (Figure 1). The analysis was organized by Dr Milko Novič and performed by Professor Detlef Günther at ETH Zurich, using Laser Ablation Inductively Coupled Mass Spectrometry (LA-ICP-MS).

Several compositional glass groups of different origins have been discerned (Figure 2). Only two samples are characterised by elevated magnesia and potash concentrations, which indicate plant ash was used as a flux for low-

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Figure 1: Samples of Novo Mesto glass included in this study.

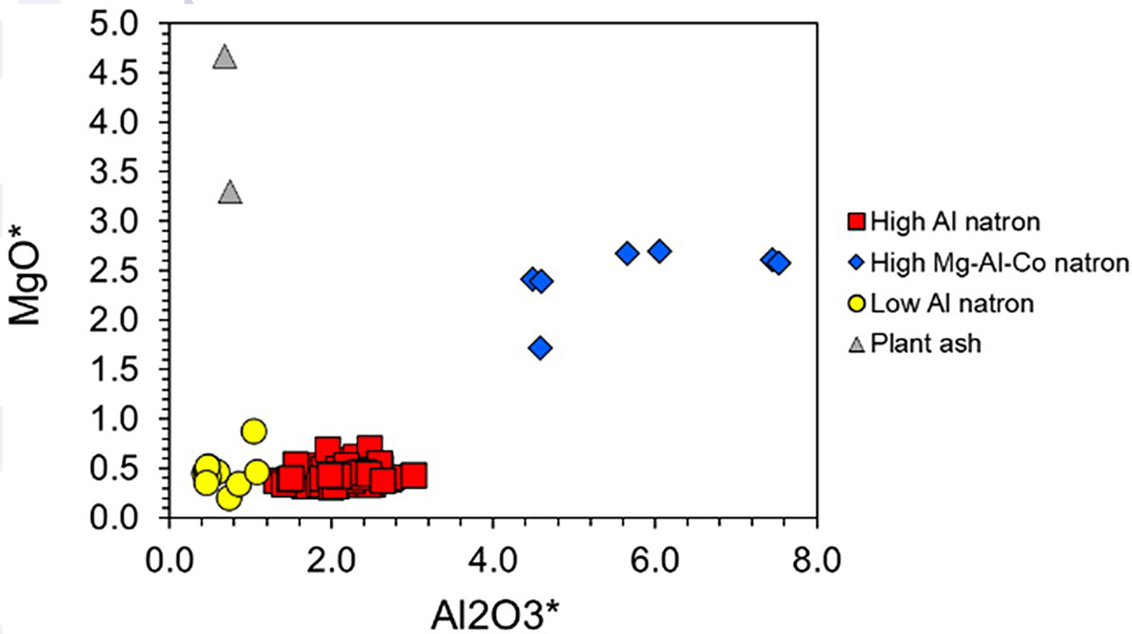

Figure 2: Main compositional types discerned in the Novo Mesto assemblage.

ering the high melting temperature of quartz (c.1710°C). The trace elements of these beads resemble those of Late Bronze Age Mesopotamian glasses from Nuzi and Tell Brak (Shortland *et al.* 2007). As this glass type is not frequent in the assemblage, it is likely an heirloom, presumably of Mesopotamian provenance, which remained in circulation.

The rest of the assemblage is characterised by low potash and magnesia concentrations, indicating that mineral natron was used as a flux. This sodium bicarbonate mineral was the prevailing flux in glasses found in the Mediterranean and Europe from the first millennium BCE well into the ninth century CE (Phelps et al. 2016; Schlick-Nolte and Werthmann 2003, 29; Shortland *et al.* 2005, 522).

These natron glasses can be further subdivided according to their alumina concentrations. As alumina enters the glass batch as an accompanying element in the silica minerals present in the sand impurities – feldspar and clay – it indicates sand origins. The occurrence of glasses with higher and lower alumina concentrations suggests at least two different sources of sand in this compositional glass type. Moreover, the differences in the amounts of zirconium and strontium further corroborate different provenance of the sands used. Zirconium is usually more abundant in inland sands, and strontium is more abundant in marine sands (Degryse *et al.* 2006, 497; Wedepohl and Baumann 2000, 129).

Glasses with lower alumina concentrations have lower strontium and higher zirconium, and most glasses with higher alumina concentrations, except for a few outliers, have lower zirconium and higher strontium concentrations. The sands used in natron glasses with higher alumina have a very similar chemical composition to Levantine sands, which were extensively used in the later, Hellenistic, and Roman glass production, and are mentioned by Pliny as a good source for glassmaking (NH 36; Eichholz 1962, 151). It appears Levantine glass was traded and produced on a larger scale before the Hellenistic period. A smaller subset of this group with elevated zirconium is possibly Egyptian in origin.

The origin of the glass with lower alumina concentrations is ambiguous. According to their trace element pattern, the earliest examples are likely of Mesopotamian origin, while the Early and Late Iron Age low alumina glasses share similarities with Egyptian sands. Another exciting possibility is that the latter could be local Italian production: a recent study of West Mediterranean sand deposits has shown that beach sands from the northeast part of the Salentina peninsula in southern Italy would produce a glass similar to standard natron glass but with lower alumina, which corresponds with these studied examples (Brems *et al.* 2012, 2902; Brems and Degryse 2014, 117). However, there is no archaeological evidence of the glass industry in the region at this time, and more research is needed before any conclusion can be reached.

Finally, four cobalt-blue glass samples, the earliest dated beads in the studied assemblage, contain high alumina and magnesia, indicating the use of a specific colourant which is attributed to cobalt-bearing alums from the western desert of Egypt. This source was first defined by Kaczmarczyk in 1986. The trace elements of these alum-coloured glasses are affected by the contamination of the strong colourant. However, the sediment-related elements somewhat resemble Mesopotamian raw materials, which can be taken as a suggestion of their origin. This type of cobalt blue glass appears to occur widely in the tenth-eight centuries BC and has been reported from Iraq (Reade *et al.* 2005) to France (Gratuze 2009).

Regarding the colouration of the beads, opaque colours are more common than translucent ones. The most common bead colour is blue, followed by yellow, white, colourless, green, and turquoise, and the most common decoration colours are white and yellow.

Cobalt, from at least three different sources, was used as the colourant for the blue glasses. Alongside the already mentioned cobalt alum, a second cobalt source is characterised by elevated arsenic and nickel concentrations. Its signature is comparable to the European Erzgebirge Mountain range cobalt ores, as well as some Iranian cobalt sources; both were actively exploited during this period of prehistory (Gratuze 2013, 323; Walton *et al.* 2012). The third cobalt source, defined by elevated copper concentrations, is analogous to the later Roman cobalt sources (Gratuze *et al.* 2018, 5). One bead was coloured very dark blue/black with the addition of iron.

Yellow glass is coloured and opacified with lead antimonate, and white glass with calcium antimonate; these compounds are typical opacifiers and colourants of the period (Shortland 2002, 519). Only a few samples are colourless. Most of these are intentionally decolourised with antimony, and one sample is naturally translucent and contains slightly elevated Cu concentrations that suggest recycling. The turquoise glass was made by adding copper to calcium antimonate white glass, and green glass was made by adding copper to lead antimonate yellow glass. In one of the samples, cobalt is present alongside copper, indicating a possibility that a yet undocumented practice of mixing cobalt blue glass with the lead antimonate yellow glasses to achieve the desired colour. Furthermore, another distinct practice – often seen in later workshops producing Roman mosaic vessels (Freestone and Stapleton 2015, 70) – of overlaying translucent copper-blue or turquoise glass over the opaque yellow glass to achieve a green hue – was detected in two samples. This suggests that the workshop that produced the beads was not colouring the raw glass but just used the available imported materials to produce various colours.

Conclusion

In conclusion, the presented Novo Mesto data indicates that glass used for bead production was imported. The trace elements of Novo Mesto glasses show analogies to the already defined glass types provenanced to the eastern Mediterranean, and natron, a geographically restricted ingredient, is the source of soda for the most significant part of the assemblage. At present, there is not much archaeological evidence of natron being traded as a raw ingredient. The results, however, reveal the existence of organised and well-supplied long-distance glass trade between the eastern Mediterranean and Lower Carniola during the Iron Age, asserting the strategic geographical position of the Novo Mesto settlement and its active participation in the prehistoric trade networks. Local glassworking workshops remain to be archaeologically confirmed, but the possibility that the glass was imported as ingots and worked locally is likely.

The reasons why glass was imported can only be hypothesized. It is likely due to the restricted sources of natron, suitable sand of a complimentary composition, as well as possibly the lack of specific know-how, but perhaps the intentional exclusivity and symbolic significance of the far-derived material was a choice employed to maintain the construction and display of local elite identities. Hopefully, future research will be able to provide new insights on glass use in Lower Carniola.

Summary

With its abundant glass bead assemblage, Novo Mesto is one of the key sites for prehistoric glass studies in Europe. No remains of glass production workshops have yet been archaeologically confirmed, but the myriad of glass items has suggested that local glassmaking workshops existed in Novo Mesto. This report presents the first quantitative trace element data on the Novo Mesto glass assemblage that offers a fresh perspective on prehistoric glass use and production. The analysed material consists of forty-eight Late Bronze Age to Late Iron Age glass beads and one Late Iron Age glass bracelet from the Kapiteljska Njiva and Mestne Njive cemeteries. Several compositional glass groups of different origins have been discerned. Only two samples are characterised by elevated magnesia and potash concentrations, which indicate plant ash was used as a flux, and the trace elements of these beads resemble those of Late Bronze Age Mesopotamian glasses. The rest of the assemblage is characterised by low potash and magnesia concentrations, indicating that mineral natron was used as a flux.

These natron glasses can be further subdivided according to their alumina concentrations. Cobalt, from at least three different sources, was used as the colourant for the blue glasses. The presented Novo Mesto data indicates that glass used for bead production was imported. The trace elements of Novo Mesto glasses show analogies to the already defined glass types provenanced to the eastern Mediterranean, and natron, a geographically restricted ingredient, is the source of soda for the most significant part of the assemblage. The results reveal the existence of organised and well-supplied long-distance glass trade between the eastern Mediterranean and Lower Carniola during the Iron Age, asserting the strategic geographical position of the Novo Mesto settlement and its active participation in the prehistoric trade networks.

Povzetek

Novo mesto je s svojimi bogatimi najdbami steklenih jagod eno najpomembnejših najdišč za študij prazgodovinskega stekla v Evropi. Dokazi o lokalni proizvodnji stekla še niso bili arheološko potrjeni, toda izjemna količina steklenega gradiva je spodbudila hipotezo o obstoju lokalnih steklarskih delavnic v Novem mestu. V tem članku predstavljamo prve podatke in rezultate naravoslovnih analiz stekla iz Novega mesta, ki nudijo svež pogled na uporabo in proizvodnjo prazgodovinskega stekla. Analizirano gradivo je obsegalo 48 pozno bronastodobnih in pozno železnodobnih jagod ter odlomek latenske zapestnice s prazgodovinskih grobišč Kapiteljska njiva in Mestne njive. Razločiti je bilo mogoče več skupin glede na sestavo stekla. Samo dva vzorca sta imela značilno povečano koncentracijo magnezija in sode, kar pomeni, da so kot topilo uporabili pepel morskih rastlin; elementi v sledovih pri teh jagodah so podobni kot pri steklo pozne bronaste dobe v Mezopotamiji. Ostali vzorci imajo značilno nizko koncentracijo magnezija in sode, kar kaže, da je bil za topilo uporabljen natron.

To skupino stekla je mogoče deliti dalje v podskupine glede na koncentracijo aluminija. Kot barvilo za temno modro steklo so uporabljali kobalt, ki kaže na tri različne izvore. Rezultati analiz gradiva iz Novega mesta tako kažejo, da je bilo steklo, uporabljeno za izdelavo jagod, uvoženo. Elementi v sledovih pri steklu iz Novega mesta kažejo analogije z že opredeljenimi tipi stekla, ki izvirajo iz vzhodnega Sredozemlja, natron, geografsko ozko omejena sestavina, pa je bil vir sode v pretežnem delu analiziranih vzorcev. Rezultati tako kažejo na obstoj organizirane in dobro vzdrževane trgovine na dolge raz-

dalje med Dolenjsko in vzhodnim Sredozemljem v času železne dobe; hkrati dokazujejo strateško geografsko lego Novega mesta in njegovo aktivno vlogo v širši trgovski mreži železne dobe.

References

Brems, D., P. Degryse, F. Hasendoncks, D. Gimeno, A. Silvestri, E. Vassilieva, and J.Honings. 2012. "Western Mediterranean sand deposits as a raw material for Romanglass production." *Journal of Archaeological Science* 39: 2897–2907.

Brems, D., and P. Degryse. 2014. "Trace Element Analysis in Provenancing Roman GlassMaking." *Archaeometry* 56: 116–136.

Degryse, P., J. Schneider, U. Haack, V. Lauwers, J. Poblome, M. Waelkens, and P. Muchez.2006. "Evidence for glass 'recycling' using Pb and Sr isotopic ratios and Sr-mixinglines: the case of early Byzantine Sagalassos." *Journal of Archaeological Science 33:*494–501.

Freestone, I.C., and C.P. Stapleton. 2015. "Composition, Technology and production ofcoloured glasses from Roman mosaic vessels". In *Glass of the Roman World*, edited by Justine Bayley, Ian C. Freestone, and Caroline Jackson, 61–76. Oxford: Oxbow Books.

Gabrovec, S. 1987. "Dolenjska grupa." In *Prahistorija jugoslavenskih zemalja*. 5 vols, editedby Alojz Benac, V, 29–119. Sarajevo: Akademija nauka i umjetnosti Bosne iHercegovine.

Gratuze, B. 2009. "Les premiers verres au natron retrouvés en Europe occidentale: composition chimique et chronotypologie." In *Annales du 17° Congrès de l'Association Internationale pour Histoire du Verre. Annales of the 17th Congress of the International Association for the History of Glass, Antwerp*, edited by Koen Janssens, Patrick Degryse, Peter Cosyns, Joost Caen, and Luc Van't dack: 8–14. Antwerp: University Press Antwerp.

Gratuze, B. 2013. "Provenance analysis of glass artefacts." In *Modern methods for analysingarchaeological and historical glass,* edited by Koen Janssens: 311–344. Chichester: Wiley.

Gratuze, B., I. Pactat, and N. Schibille. 2018. "Changes in the Signature of Cobalt Colorantsin Late Antique and Early Islamic Glass Production." *Minerals* 8: 1–20.

Haevernick, T.E. 1974. "Zu den Glasperlen in Slowenien." *Opuscula Iosepho Kastelic Sexagenario Dicata, Situla* 14/15: 61–65.

Kaczmarczyck, A. 1986. "The source of cobalt in ancient Egyptian pigments." *Proceedings ofthe 24th International Symposium on Archaeometry [held at the Smithsonian Institution, Washington D.C., from 14 to 18 May 1984],* edited by Jacqueline S. Olin: 369–76. Washington: Smithsonian Institution Press.

Križ, B. 2009. "The Early Iron Age (8th–4th century BC)." In *The Archaeological Image ofDolenjska,* edited by Borut Križ, Petra Stipančić, and Andreja Škedelj Petrič: 87–141 Novo Mesto: Dolenjski Muzej,

Križ B., and M. Guštin. 2015. "Prehistoric glass from Novo mesto/ Slovenia." In *Annales du 19e Congrès de l'Association internationale pour l'histoire du verre: Piran 2012,*edited by Irena Lazar, 48–54. Koper: AIHV.

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

Pliny The Elder. 1962. *Natural History. Books 3-4.* Translated by D. E. Eichholz, edited by Rackham, H., W.H.S. Jones, and D.E. Eicholz, *36-37.* Cambridge, MA: Harvard University Press, Loeb Classical Library.

Reade, W.J., I.C. Freestone, and S.J. Simpson. 2005. "Innovation or continuity? Early first millennium BCE glass in the Near

East: the cobalt blue glasses from Assyrian Nimrud." *Annales Du 16e Congres de l'Association Internationale Pour l'Histoire Du Verre, London, 2003. Nottingham.* AIHV: 23–27.

Schlick-Nolte, B., and R. Werthmann. 2003. "Glass Vessels from the Burial of Nesikhons." *Journal of Glass Studies* 45: 11–34.

Shortland, A. 2002. "The use and origin of antimonate colourants in early Egyptian glass." *Archaeometry* 44: 517–530.

Shortland, A., L. Schachner, I.C. Freestone, and M.S. Tite. 2005. "Natron as a flux in the early vitreous materials industry: Sources, beginnings and reasons for decline." *Journal of Archaeological Science* 33: 521–530.

Shortland, A., N. Rogers, and K. Eremin. 2007. "Trace element discriminants between Egyptian and Mesopotamian Late Bronze Age glasses." *Journal of Archaeological Science* 34: 781–789.

Walton, M., K. Eremin, A. Shortland, P. Degryse, and S. Kirk. 2012. "Analysis of Late Bronze Age glass axes from Nippur- a new cobalt colourant." *Archaeometry* 54: 835–852.

Wedepohl, K. H., and A. Baumann. 2000. "The Use of Marine Molluscan Shells for Roman Glass and Local Raw Glass Production in the Eifel Area (Western Germany)." *Naturwissenschaften* 87: 129–132.