Lead Isotope Analysis of Slag-tempered Negev Highlands Pottery

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Summary: Lead Isotope Analysis of Slag-Tempered Negev Highlands Pottery
Petrographic analysis of Iron IIA Negev Highlands pottery revealed that the clay used in some of the vessels was tempered with copper smelting slag. Here we show, using lead isotope analysis, that the slag was likely a byproduct of the contemporaneous smelting operations at Faynan, Jordan. We substantiate previous observations regarding the connection between settlements in the Negev Highlands and the mining and smelting operations in Wadi Arabah.

Article received: July 6th 2015; approved: September 8th 2015.

Keywords: Iron IIA – Negebite pottery – Slag temper – Negev Highlands – Wadi Faynan – Timna – Lead isotope analysis

Resumen: Análisis de isótopos de plomo de cerámica de las tierras altas del Negev atemperada con escoria
El análisis petrográfico de la cerámica de las tierras altas del Negev de la Edad del Hierro IIA reveló que la arcilla utilizada en algunos recipientes fue templada con escoria resultante de la fundición de cobre. En este trabajo mostramos, utilizando análisis de isótopos de plomo, que la escoria era probablemente un subproducto de las operaciones de fundición contemporáneas en Feinán, Jordania. Corroboramos observaciones previas con respecto a la conexión entre asentamientos en las tierras altas del Negev y las operaciones mineras y de fundición en el Wadi Arabá.

Palabras Clave: Edad del Hierro IIA – Cerámica negevita – Templado de escoria – Tierras altas del Negev – Wadi Feinán – Timna – Análisis de isótopos de plomo

Introduction

Recent petrographic analysis of wheel- and handmade ('Negebite') pottery found at early Iron IIA Negev Highlands sites has established the connection of these settlements with the copper extraction centers in Wadi Arabah (**Fig. 1**).[1] Most significantly, a group of (almost exclusively handmade) vessels was made of clay which was tempered with minute fragments of crushed slag, characterized as copper smelting slag with the aid of metallographic and scanning electron microscopes. A production of these vessels in Wadi Arabah was proposed. Here we report the results of lead isotope analysis of slag inclusions in selected vessels from the Negev Highlands sites. The study provides additional evidence for the origin of this pottery in the Arabah mining districts, most likely Wadi Faynan.

Archaeological and Historical Context

Crushed slag is available in the form of extensive heaps at the Arabah smelting sites, and indeed slag tempering is a well-known phenomenon in this region in the Iron Age, both in domestic and in refractory ceramics.[2]

[1] Martin and Finkelstein 2013; Martin *et al.* 2013.
[2] Slatkine 1978: 114; Rothenberg 1980: 198; 1988: 201; Bachmann and Rothenberg 1980: 220; Glass 1988: 103, 109–111; Tite *et al.* 1990; Al-Shorman 2009: 203–223; Smith 2009: 365–417 [Wares A2, A2b and A6b], 572; 2014; Ben-Yosef 2010: 364, 674–676, 691, 699, 702–703.

The production of slag-tempered Negev Highlands vessels in Wadi Arabah is not only substantiated by the slag inclusions but also by certain rock and mineral fragments encountered in the fabrics. These include argillaceous shales, sandstones, coarse-very coarse angular quartz and, most importantly, intrusive and volcanic igneous rock fragments, mainly of felsic (granite, rhyolite) and, rarely, also of intermediate composition (diorite, andesite). This temper assemblage can be sourced to the southerly desert regions of southern Jordan, the eastern and southern Arabah and the southern Sinai, where rocks of the Precambrian crystalline basement of the Arabian-Nubian Shield and of the Paleozoic-Late Mesozoic continental environs outcrop extensively.[3] Combining geological with archaeological considerations, Wadi Arabah and, more precisely the copper districts of Wadi Faynan and Timna—the only focus of human activity in this region during the Iron IIA—remain the only viable candidate.[4]

It is futile to consider the option that crushed slag was transported to the Negev Highlands separately, to be added to locally procured clays.[5] Such a scenario would also require the 'import' of other tempering agents (see above), such as granitic rocks, for which a local Negev Highlands origin can be categorically excluded. Ethnographic studies have shown that in traditional pottery production, raw clay and temper are generally not transported over distances more than *ca*. 10 km.[6] Moreover, no evidence of pottery kilns has ever been recorded in the Negev Highlands.[7]

On the basis of shape repertoire—almost exclusively the most essential, open household types (mostly cooking vessels)—and production mode ("household production") it was argued that the handmade wares were not prone to be exchanged as trade items, but reached the Negev Highlands as a result of movement of people; it was brought by its owners, who "commuted" between the Negev Highlands and Wadi Arabah.

Both the Negev Highlands and the copper districts in Wadi Arabah were dominated by a nomadic milieu with tribal organization.[8] In the early Iron IIA

[3] For a detailed discussion and references, see Martin and Finkelstein 2013: 24–27; Martin *et al.* 2013: 3780–3781, 3786–3787.

[4] In the Iron IIA no evidence of settlement activity has been found on the southern Jordan plateau (Bienkowski 1992a, 1992b; Herr and Najjar 2001). A near settlement vacuum was encountered in the Sinai during the entire Iron II (Meshel 2002: 287; Yezerski 2003).

[5] Martin *et al.* 2013: 3787.

[6] Arnold 1985: 32–60; cf. Goren, Finkelstein and Na'aman 2004: 6–7.

[7] Haimann and Goren 1992: 149.

[8] Fantalkin and Finkelstein 2006; Tebes 2006; Levy 2009a; 2009b; Ben-Yosef 2010: 648–656; for the Wadi Fidan 40 cemetery, see Beherec, Najjar and Levy 2014.

pastoral nomadic groups in the Negev Highlands were in a process of sedentarization. Different lines of data suggest that at least some of these groups were involved in the Arabah copper production system—as miners and smelters. They may have also been involved in the transportation of copper, likely in ingot form, from the mines to northern urban centers and to the Mediterranean shore, where the copper could have been loaded onto ships. The latter possibility was raised by the chemical and isotopic analyses of ingots from a cargo comprising 54 loaf-shaped copper ingots retrieved from the Carmel coast, near Neve Yam, suggesting that the ingots were made of copper from Wadi Arabah ores, specifically those at Faynan.[9]

The Negev Highlands population was an integral part of a prosperous network propelled by the profitable copper market. This network included Wadi Arabah, which after the disruption of the Cypriot trade at the end of the Late Bronze Age rose as the main copper provider of the southern Levant, the Beer-sheba Valley, the Negev Highlands and the Mediterranean coast. The economic boom in the south stimulated the sedentarization process in the Negev Highlands.[10] Control of this network must have been one of the main goals of the Sheshonq I campaign to the southern Levant.[11]

In the period discussed here, the leading copper producer in the Arabah was the Wadi Faynan district, with the largest production site located at Khirbet en-Nahas.[12] On a smaller scale, mining and smelting activity also occurred in the Timna area,[13] arguably under the same production system and operated by the same groups.[14]

From a chemical analysis it emerged that the crushed slag added to the ceramic wares as tempering agent is manganese-rich and often contains an appreciable amount of phosphorus (P_2O_5).[15] This composition points to the Cambrian Dolomite-Limestone-Shale (DLS) unit as host rock for the mined

[9] Yahalom-Mack *et al.* 2014.

[10] A somewhat similar process can be identified in the Early Bronze Age III-Intermediate Bronze Age. For copper-related activities in this period in Wadi Faynan, see Levy *et al.* 2002. The contemporaneous subsistence and settlement pattern in the Negev Highlands is currently under investigation by our team (see already Dunseth 2013).

[11] Fantalkin and Finkelstein 2006; Finkelstein and Piasetzky 2008.

[12] E.g., Levy *et al.* 2005; 2014.

[13] E.g., Ben-Yosef 2010: 507–621; Ben-Yosef *et al.* 2012.

[14] Ben-Yosef 2010: 955–959.

[15] Martin *et al.* 2013: 3787–3788.

copper.[16] The DLS unit was the primary source for copper ore in Wadi Faynan (the local Burj formation), where it is widely exposed. In Timna, most of the copper-bearing Cambrian ores (the local Timna formation in particular) do not outcrop on the surface and, instead, the iron-rich sandstones of the Cretaceous Amir and Avrona formations were exploited for copper throughout all periods of activity. There is, however, limited evidence that during the Iron IIA the miners of the Timna Valley have also used the Cambrian manganese-rich ores (Layer I at Site 30; 9th century BCE).[17]

Materials and Methods

Four slag-tempered vessels, each from a different Negev Highlands site, were selected for lead isotope analysis (**Table 1**; for the location of sites, see **Fig. 1**; for the illustration of the vessels, see **Fig. 2**; for micrographs of fabric inclusions, see **Figs. 3–5**). This method is based on the fact that no isotope fractionation occurs during the copper smelting and re-melting processes. The lead isotope ratios thus serve as a 'fingerprint' of the mineral ore deposits, which can be compared with the end-product.[18]

Fig.	*Site*	*Reg. Nº*	*Vessel type*	*Reference*	*Fabric and inclusions*
2: 1	Refed	5/1	Cooking krater	Meshel and Cohen 1980: Fig. 3: 4	Silty, non-calcareous, micaceous clay + slag, quartz, sandstone, quartzite(?)
2: 2	Atar Haroʿa	84/2	Cooking(?) krater	Cohen 1970: Fig. 11: 13	Calcareous clay + slag, calcareous sand, quartz, vegetal temper
2: 3	Horvat Ritma	53/1	Lamp	Meshel 1977: Fig. 7: 10	Silty, non-calcareous, micaceous clay + quartz, granite, slag, andesite, feldspar, sandstone
2: 4	Ramat Matred	1216/1	Cooking krater	Cohen and Cohen-Amin 2004: Fig. 40: 13	Silty, non-calcareous, shaley clay + slag, quartz, limestone

Table 1.
Slag-tempered Negev Highlands vessels sampled for this study.

[16] Cf. Hauptmann 2007: 63–79; Ben-Yosef 2010: 96–104; Levy, Ben-Yosef and Najjar 2014: 12–21.
[17] Tite *et al.* 1990; Ben-Yosef 2010: 564–571, 901–903; Ben-Yosef *et al.* 2012.
[18] For discussion and bibliography, see Gale and Stos-Gale 1982; Hauptmann 2007: 31–38; Stos-Gale and Gale 2009; Pernicka 2014.

A 2–3 cm vessel fragment was crushed lightly and pieces of slag, visible to the naked eye, were collected. The slag fragments with some adhering clay were dissolved in a mixture of hydrofluoric and nitric acid and diluted for chemical and isotopic analysis. Lead concentration was determined using a quadrupole Inductively Coupled Plasma – Mass Spectrometer (ICP-MS, Agilent 7500cx). Following the separation of Pb in columns lead isotopic ratios were measured using Neptune plus multi-collector ICP-MS. Thallium was used for mass-bias correction. SRM-981 standard was run with the samples yielding the following values: $^{208/206}$Pb = 2.1660±3.6E^{-05}, $^{207/206}$Pb = 0.9145±1.4E^{-05}, $^{204/206}$Pb = 0.0591±6.0E^{-06}.

Results and Discussion

The results are presented in **Table 2** and are plotted in **Fig. 6** against the lead isotope ratios of the following ores and artefacts:

1 Ores from the DLS at Faynan (Burj formation), which was the main mineralization exploited during the Bronze and Iron Ages;[19]
2 Slag fragments from Khirbet en-Nahas, Wadi Dana and Feinan 5, which are dated to the Iron Age;[20]
3 Sandstone ores from Timna (the Cretaceous Amir and Avrona formations), which were the main Cu-hosting rocks exploited in this region;[21]
4 Pb-rich Mn nodules (Type B) from the Cambrian Timna Formation, which are equivalent to the DLS ores at Faynan. These do for their most part not outcrop in the Timna Valley and were generally not exploited in ancient times;[22]
5 Ingots from the Early to Intermediate Bronze Age metallurgical activity at Khirbet Hamra Ifdan, Wadi Faynan area;[23]
6 Late Bronze Age ingots from Timna;[24]
7 Ingots from the Carmel coast near Neve Yam.[25]

[19] After Hauptmann *et al.* 1992.
[20] Hauptmann *et al.* 1992.
[21] Gale *et. al.* 1990; Segev, Beyth and Bar-Matthews 1992; Hauptmann *et al.* 1992; Hauptmann 2007; Asael *et. al.* 2012.
[22] Ehrlich *et al.* 2004.
[23] Levy *et al.* 2002; Hauptmann *et al.* 2015.
[24] Yahalom-Mack *et al.* 2014.
[25] Yahalom-Mack *et al.* 2014.

Figure	*Site*	$^{208}Pb/^{206}Pb$	*e*	$^{207}Pb/^{206}Pb$	*e*	$^{204}Pb/^{206}Pb$	*e*
2: 1	Refed	2.1214	0.00002	0.8707	0.000006	0.0557	0.000002
2: 2	Atar Haroʿa	2.1184	0.00002	0.8691	0.000008	0.0556	0.000001
2: 3	Horvat Ritma	2.1192	0.00002	0.8699	0.000007	0.0557	0.000002
2: 4	Ramat Matred	2.1195	0.00003	0.8700	0.000009	0.0557	0.000002

Table 2.
Lead isotope ratios of slag from Negev Highlands vessels sampled for this study.

Figure 6 shows that the slag fragments from the Negev Highlands vessels are consistent with both Timna and Faynan copper ores. However, the major source of exploited ore from Timna (i.e., the Cretaceous Amir and Avrona formations) has a large scatter, while the Faynan ores plot very close to the sampled vessels. In addition, the vessels are fully consistent with the crescent-shaped ingots from the Early Bronze Age III and Intermediate Bronze Age metallurgical activity at Khirbet Hamra Ifdan, which utilized the DLS ores in this region, as well as with the slag from the Iron Age smelting sites at Faynan and with the ingots from Neve Yam. This correspondence and the archaeological data showing that the DLS ores were extensively exploited at Faynan during the Iron Age suggests that the slag in the Negev Highlands vessels likely originated in this region. Only eight samples from the DLS unit at Faynan were analyzed for their lead isotope ratios[26] and interestingly, most of them cluster slightly lower than slag and ingot samples. This difference may be bridged with additional analyses.

The particular member within the Timna formation which is geochemically equivalent to the Faynan DLS unit appears to be the lead-rich Type B manganese nodules within the Timna Formation. It should be, however, noted that the lead isotope ratios of these nodules are not identical to those of the Faynan DLS unit, as should be expected (**Fig. 6**). This difference may be an artifact of the small number of samples obtained from these units. Otherwise, this may point to a slight geochemical difference between these formations.

Since in the Timna Valley Cambrian manganese-rich ores of the Timna formation were used during the Iron IIA in addition to ores derived from Cretaceous sandstones (see above), theoretically they could have been the source of the slag in the Negev Highlands vessels. However, the perfect consistency of the lead isotope ratios of the slag temper with the Faynan ingots suggests that Faynan is indeed the source of the slag added to the Negev Highlands ceramics.

[26] Hauptmann *et al.* 1992.

This is supported by the geographical proximity of Wadi Faynan and the Negev Highlands and by the fact that in the Iron IIA the Faynan mines were exploited at an incomparably grander scale than those at Timna.

Conclusion

The results of the lead isotope analysis of slag fragments extracted from Negev Highlands vessels substantiate previous observations, based on archaeological considerations, petrographic analysis and mineralogy of the slags, regarding the connection between the Negev Highlands Iron Age settlements and the mining and smelting operations in Wadi Arabah. This suggests that Iron IIA sedentary activity in the Negev Highlands was related to the thriving metallurgical activities at Wadi Faynan. The economic boom in the southern desert regions stimulated the sedentarization process in the Negev Highlands. We can reconstruct a scenario in which some of the Negev Highlands pastoral nomadic groups periodically worked in the Wadi Arabah copper districts as miners and smelters, probably in order to supplement their subsistence economy.

Acknowledgments

This study was partially funded by the European Research Council under the European Community's Seventh Framework Program (FP7/2007–2013)/ERC grant agreement no. 229418. It was part of a project headed by Israel Finkelstein, Tel Aviv University and Steve Weiner, Weizmann Institute of Science. Additional funding was provided by the Lady Davis Foundation.

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Figures

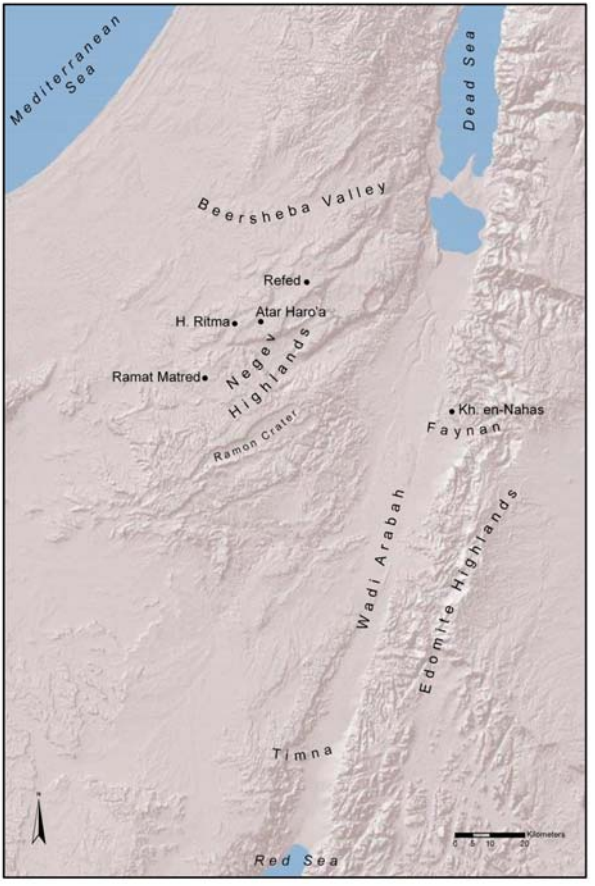

Fig. 1.
Map of southern Israel and Jordan showing the main regions and sites mentioned in the article.

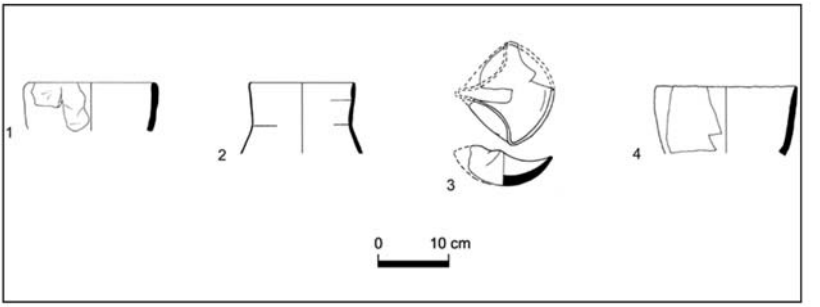

Fig. 2.
Selected slag-tempered Iron IIA Negev Highlands pottery, including handmade Negebite (cooking) kraters (1, 2, 4) and a wheel-made lamp (3). Courtesy of the Israel Antiquities Authority and the Institute of Archaeology of Tel Aviv University.

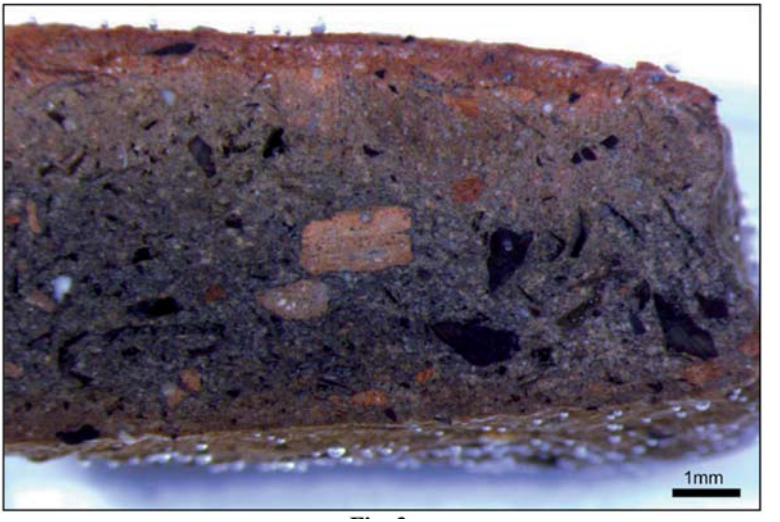

Fig. 3.
Fresh break of slag-tempered ware in the stereo-microscope (from vessel in **Fig. 2: 2**). The slag appears in the form of dark angular inclusions.

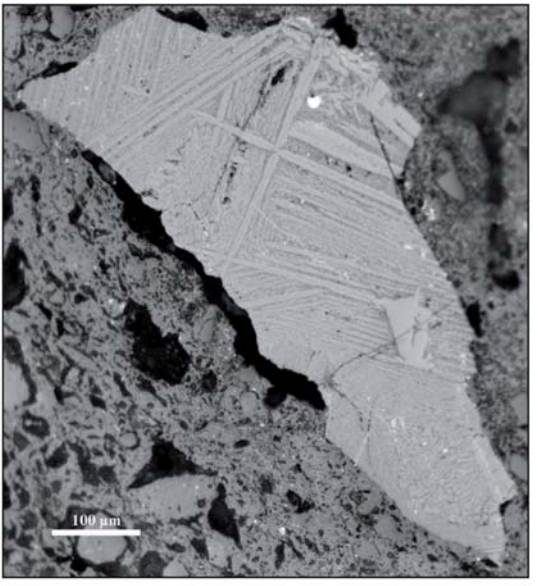

Fig. 4.
Micrograph (reflected light mode) showing fragment of crushed copper smelting slag added as temper into the clay mass of a handmade Negebite vessel (**Fig. 2: 4**).

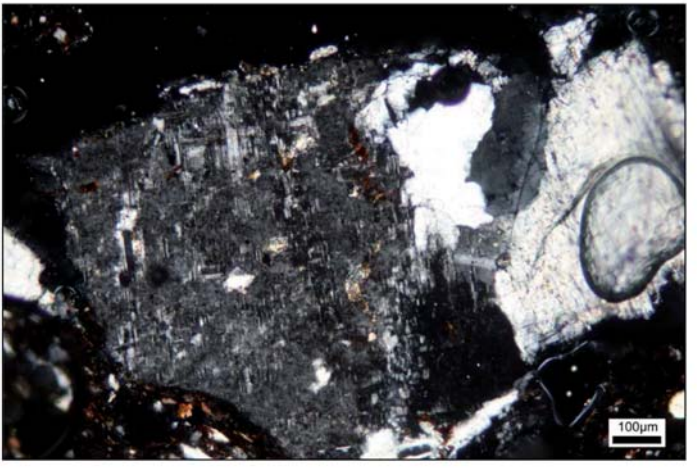

Fig. 5.
Micrograph (polarizing microscope, crossed polarized light) showing granite inclusion in the fabric of a lamp (**Fig. 2: 3**).

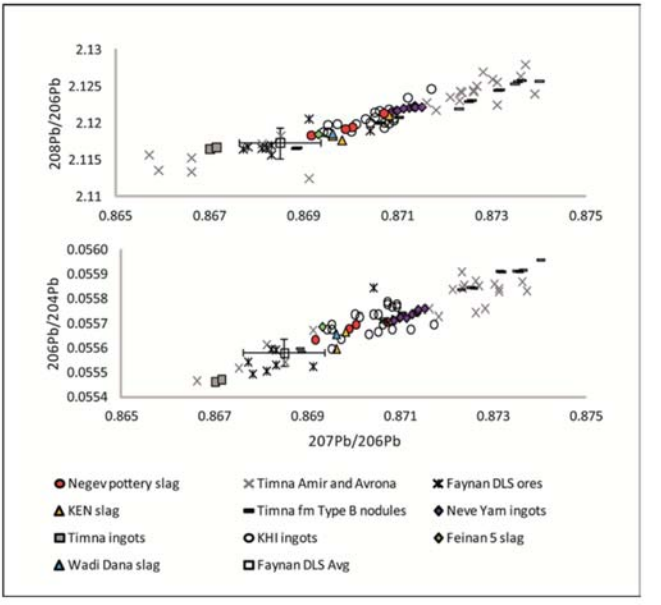

Fig. 6.
Lead isotope ratios of slag fragments analyzed for this study (**Table 2**), plotted against selected ores, ingots and slag from Wadi Arabah (for references, see text). KHI=Khirbet Hamra Ifdan, KEN=Khirbet en-Nahas.