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THE PROVENANCE OF OBSIDIAN ARTEFACTS FROM THE WĀDĪ ATH-THAYYILAH 3 NEOLITHIC SITE (EASTERN YEMEN PLATEAU) BY LA–ICP–MS*

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The geological sources of obsidian in the Red Sea region provide the raw material used for the production of obsidian artefacts found in prehistoric sites on both sides of the Red Sea, as far afield as Egypt, the Persian Gulf and Mesopotamia. This paper presents the chemical characterization of five obsidian geological samples and 20 prehistoric artefacts from a systematically excavated Neolithic settlement in highland Yemen. The major element concentrations were determined by SEM–EDS analysis and the trace element concentrations were analysed by the LA–ICP–MS method, an almost non-destructive technique capable of chemically characterizing the volcanic glass. A comparison of archaeological and geological determinations allows the provenance of the obsidian used for the Neolithic artefacts to be traced to definite sources in the volcanic district of the central Yemen Plateau.

KEYWORDS: OBSIDIAN, TRACE ELEMENTS, SEM–EDS, LA–ICP–MS, YEMEN PLATEAU, NEOLITHIC EXCHANGE

INTRODUCTION

In prehistory, obsidian was used as a choice raw material for stone tools, with a greater frequency in regions possessing or connected with dependable sources. Along with limited rates of indifferent utilization, or the occasional employment for luxury objects, obsidian was normally preferred in connection with its chief attributes of fracture predictability and exceptional cutting-edge quality. Selective use of obsidian for the making of specialized chipped-stone tools was particularly common in many periods and cultures, including the production of sharp-edged, microlithic elements for composite tools, such as documented in Yemen (e.g., Rahimi 1987; Fedele and Zaccara 2005; Khalidi 2009). Regionally, time depth was also important. In southern Arabia and the Horn of Africa, the utilization of obsidian continued well into the historical period, making this a material of prominent interest. When geometric microliths appeared in this general area, starting with the third millennium BC (Khalidi 2009), once again this production extensively, or even exclusively, relied on obsidian. However, a detailed diachronic perspective of obsidian

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exploitation and use has just begun to take shape, not only in the above-mentioned areas but throughout the Red Sea Rift region.

Researchers studying obsidian finds from archaeological contexts, and identifying the sources of obsidian employed for artefacts, can develop and test models concerning prehistoric interactions, access to resources, and trade. The considerable number of potential obsidian sources, combined with the scarcity of work aimed at the characterization of outcrops in the Red Sea Rift region, have often made it difficult to determine the provenance of obsidian finds from archaeological sites on both sides of the Red Sea, as far afield as Egypt (e.g., Bavay *et al*. 2000), the Persian Gulf and Mesopotamia. Previous studies by Zarins (1989, 1990) and Francaviglia (1985; 1990a,b; 1996) demonstrated the geochemical and geological difficulties of establishing the precise provenance of a number of obsidian archaeological finds from this general region (for early reviews, see also Overstreet *et al*. 1988, 373–91; Overstreet and Grolier 1988, 465–6; Overstreet and Grolier 1996, 350, 386–8). More recently, continuing work by L. Khalidi and colleagues as part of the VAPOR project (Khalidi *et al*. 2010; Lewis *et al*. 2010) has provided an important contribution to the knowledge of the obsidian sources in the Yemen highlands on the basis of LA–ICP–MS analyses.

Among the different analytical techniques, the LA–ICP–MS method is particularly valuable for the compositional characterization of archaeological obsidian, because it combines micro-destructivity with the capacity for analysing a great number of trace and rare earth elements with high sensitivity in a very short time. These characteristics make LA–ICP–MS a very powerful tool for the characterization and provenance determination of archaeological specimens (Gratuze 1999; Bavay *et al.* 2000; Carter *et al.* 2006; Barca *et al*. 2007; Giussani *et al*. 2009; Khalidi *et al*. 2010).

In order to expand the geochemical database of obsidian sources and compositions for the Red Sea region (Francaviglia 1990b; Khalidi *et al*. 2010), in the present study we report the results of analyses jointly carried out on geological samples and archaeological specimens from highland Yemen. The geological obsidian was collected 30 years ago from outcrops at Jabal Isbīl[1] and Jabal al-Lisī, two volcanoes in the Dhamār-Radā' volcanic field of the central Yemen Plateau, made the object of Italian volcanological studies by Chiesa *et al*. (1983); these samples were located and kindly provided by L. Lirer (University of Naples 'Federico II') and L. La Volpe (University of Bari 'Aldo Moro'). Concurrent analyses were performed on 20 obsidian artefacts from buried archaeological contexts at Wādī ath-Thayyilah 3 (WTH3), a mid-Holocene prehistoric site located on the eastern Yemen Plateau (Fig. 1). Both the geological and archaeological samples were analysed by SEM–EDS and LA–ICP–MS methods, and our geological and archaeological results were compared to the available database from the literature, with the aim of determining the provenance of archaeological specimens.

The new data presented in this paper aim to contribute, in particular, to our growing understanding of highland resource acquisition during the Holocene prehistory of Yemen. Relevant to such an aim is the excavated and dated context from which the obsidian artefacts that were sourced—comprising debitage and tools—were extracted. This opportunity to link a sourced obsidian sample to secure archaeological context represents a rare occurrence in Yemen, both from the angle of obsidian studies and prehistoric archaeology in general. These data should help better to situate in time recently published research on exchange mechanisms in prehistoric Yemen, and in the highland zone in particular (Khalidi *et al*. 2010, with references), but also the

[1]The Arabic place-names in this paper will be written in a simplified transliteration with diacritic signs omitted, partly following usage as accepted in recent obsidian research (e.g., Khalidi *et al*. 2010); and for Wādī ath-Thayyilah as 'normalized' usage in the style of Tiede (1996, xxxvi). Full transliterations can be found in several of the more archaeological- or historical-orientated references.

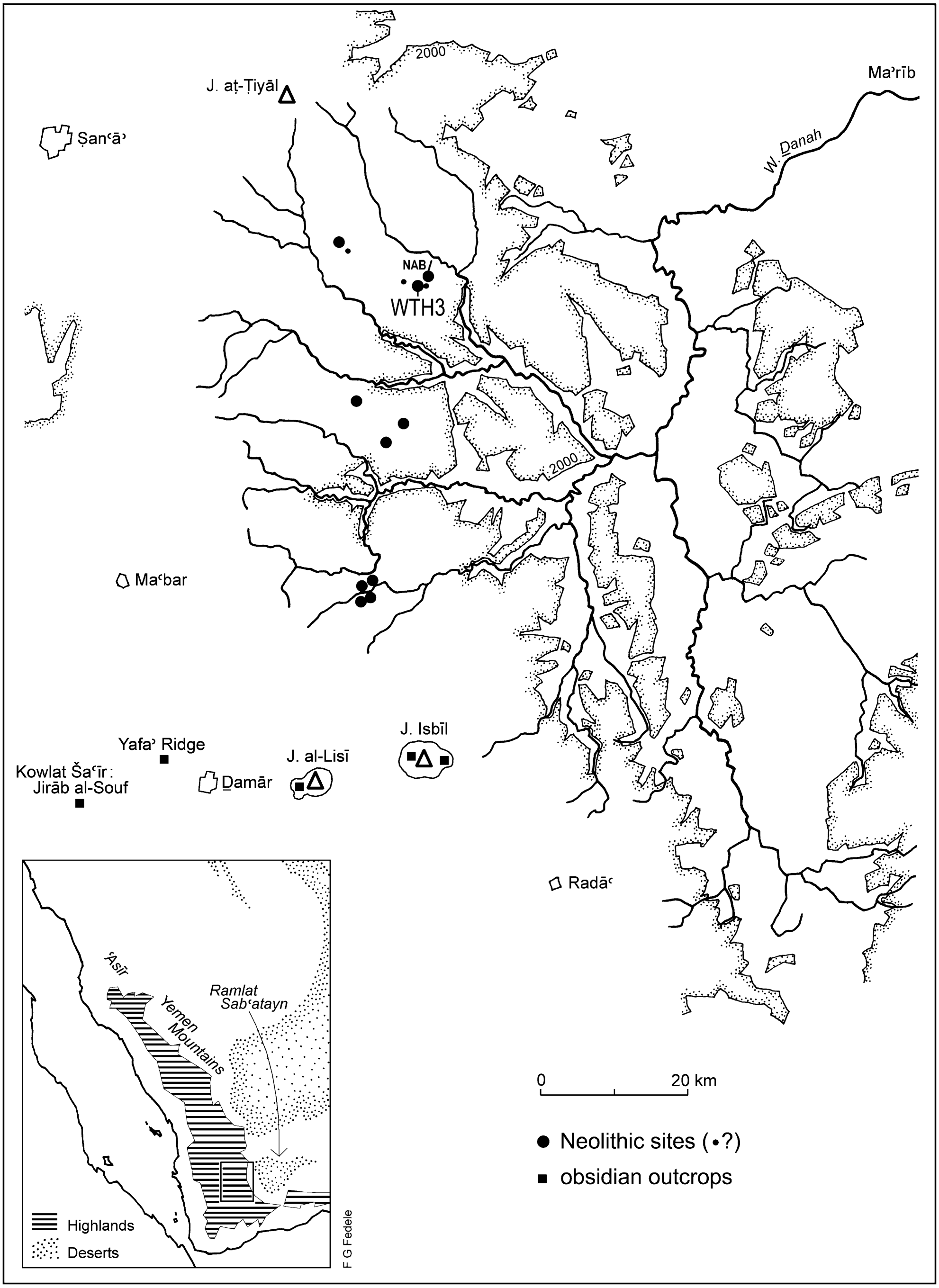

Figure 1 *A map of the eastern and central Yemen Plateau, with indications of the main Neolithic sites and known obsidian outcrops: NAB, Wādī an-Najd al-Abyad; WTH, Wādī ath-Thayyilah site 3.*

coastal Tihamah (Khalidi 2009, 288–9). Previous research (e.g., Zarins 1989, 1990; Francaviglia 1990b, 1996) had in fact been conditioned by insufficient archaeological and chronological control on chemically sourced artefactual obsidian. The WTH3 data, in addition to the geological and geochemical aspects, represent a further step towards the identification of intra-regional trade networks (specifically obsidian) and patterns of site interaction. Such subjects still need elucidation throughout southwestern Arabia.

THE WĀDĪ ATH-THAYYILAH 3 SITE AND ITS CONTEXT

WTH3 (44°39′58″ E, 15°10′00″ N) is a stratified site located in the middle–upper basin of the wadi of that name in the region of Khawlān at-Tiyāl, on the eastern Yemen Plateau, at an altitude of 2025 m. Wādī ath-Thayyilah and a fossil furrow nearby, Wādī an-Najd al-Abyad (NAB in Fig. 1), form a relatively self-contained district with a record of dense prehistoric settlement until the collapse of agricultural capacity in the second millennium BC (Marcolongo and Palmieri 1986, 1990; de Maigret 1990, 11–28; Fedele 1990a; Fedele 2009, 217–27). The site was discovered in 1983 by A. de Maigret and his team during survey work of the Italian Archaeological Mission to Yemen (de Maigret 2009, 120–6, 135–8), and then analytically investigated by one of the authors, with excavations conducted between 1984 and 1986 (Fedele 1986, 2008; Fedele and Zaccara 2005; see also de Maigret *et al*. 1988; Edens and Wilkinson 1998, 63–5).

The site is mainly defined by a Neolithic occupation, 90 × 70 m in size and extending over an estimated area of about 5300 m^2. Different types of stone features were visible on the surface, including alignments and oval or elliptical structures ('huts') made with large stone blocks. The excavations revealed a number of lighter features and hollows, both inside and outside the presumed dwelling structures. WTH3 is only one of a number of open-air settlements generally located on gentle slopes not far from water sources, and often surrounded by rich dispersals of chipped-stone artefacts. Due to a recurrent association with domestic animals, together with a lack of pottery, these highland occurrences can be characterized as representative of an aceramic Neolithic tradition.

At WTH3, as elsewhere in the Khawlān, the Neolithic occupation is linked to the so-called 'Thayyilah Palaeosol', the local expression of a particular mollisol representing a pedostratigraphic marker over a wide area of southwestern Arabia (Fedele 1986; de Maigret *et al*. 1989; Overstreet and Grolier 1996, 363–5, 372–8; Wilkinson 1997, 840–54; French 2003; Wilkinson 2003, 157–61 [the 'Jahrān soil']; Parker *et al*. 2006; Fedele 2009, 217–22). In the Thayyilah–NAB district, radiocarbon dating of the organic acids contained in this soil yielded dates of 5750 ± 500 BP—near the WTH3 site itself—and 6595 ± 75 BP (de Maigret *et al*. 1989; Marcolongo and Palmieri 1990; Fedele 2009, fig. 3), which at 2σ calibrate, respectively, to 5721–3635 BC and 5662–5382 BC.[2] This confirms the association of this soil with the mid-Holocene interval, and suggests for the eastern Plateau Neolithic a placement in the sixth to fourth millennium BC range (Fedele 2009, 221, 224; fig. 8). This dating is consistent with the techno-typological traits of the lithic assemblages, among which ovate bifacial tools and a few other foliate types, although rare (Fedele and Zaccara 2005, figs 5, 18, with typological synopsis; Fedele 2008, fig. 8 g), represent a recognizable interregional marker (e.g., Edens 1988; Edens and Wilkinson 1998, 62–4).

In the Thayyilah–NAB district, geomorphological and palynological studies yielded evidence of a mid-Holocene landscape rich in water and vegetation (Fedele 2009, 217–22, fig. 5; with

[2]Calibration according to OxCal v4.1.7 (Bronk Ramsey 2010), using the IntCal09 curve (atmospheric data from Reimer *et al*. 2009). Combined calibration of the two measurements would produce a 2σ value of 5640–5376 BC.

references); for taphonomic reasons, however, plant remains are almost completely lacking both at WTH3 and in the area. The presence in the WTH3 lithic assemblages of segmented blades that can be interpreted as sickle inserts (Fedele 2008, fig. 8 e) would suggest the importance of agricultural activities. More direct information concerning the economy of these Neolithic groups comes from the zooarchaeological evidence. The faunal collection recovered from WTH3 points to a prevalent cattle-breeding economy, with 73% of bone remains representing domestic cattle (*Bos taurus*), 16% domestic caprines (sheep and goats), and the remaining 11% wild equids and gazelles (Fedele 1990a; Fedele 2008, 164–7).

Testing below the Neolithic levels revealed a stratigraphic sequence about 1 m deep, in which evidence of earlier cultural horizons could be recognized. The best defined such horizon ('Pre-Neolithic') can tentatively be associated with a non-residential exploitation of the area during the seventh millennium BC, on the basis of an unfired clay figurine and the apparent lack of domesticated livestock (Fedele 2009, fig. 11). The stone tool assemblages coming from the Pre-Neolithic and the Neolithic horizons show many similarities and can be considered as subsequent expressions of a single cultural continuum (Fedele 2008, 164; Fedele 2009, 222–6). An obsidian blade fragment retrieved from an apparently secure Pre-Neolithic level, made from a 'grey' variety that is rare in the local Neolithic (Fedele 2009, 225, fig. 12), would predate the beginnings of obsidian use on the Tihamah (sixth millennium BC; Khalidi 2009, 281, 284). The subject will be taken up again in the last section of this paper.

The Neolithic assemblages revealed by WTH3 and several Khawlān sites are sufficiently different from those of other parts of Yemen—and especially the lowlands—to be assigned to a non-bifacial industry, a contrast that led to the hypothesis of a regional group specifically adapted to the highlands (an 'Upland Neolithic tradition'; Fedele 1986; Fedele 1990a; Edens and Wilkinson 1998, 62; Fedele 2008, 164, 167; Fedele 2009, 234). In terms of flexibility in lithic technology and land-use efficiency, this marked a successful and important period in the Yemen highland Neolithic, possibly representing the climax of a long-developed tradition. Subsequent landscape alterations generated by an interplay of tectonics and climate ended this culture, and during the third millennium BC the plateau saw the formation of an original Bronze Age—a different economy, based on caprines, and a society with different exterior relationships (de Maigret 1990; Fedele 1990a; Edens and Wilkinson 1998, 71–92). It is against this background of florescence and change that the data from Neolithic WTH3 and its allied sites, concerning obsidian procurement and highland lithic production, become significant.

WTH3: THE OBSIDIAN SAMPLE

Twenty obsidian artefacts belonging to the WTH3 Neolithic collection are archaeologically described and geochemically analysed in the present study (Table 1 and Fig. 2). The descriptive criteria and the classification follow Tixier's (1963) and Close's (1980, 1989) typologies for North African Epipalaeolithic and Neolithic assemblages, with minor adaptations as justified by the sample. The artefacts were extracted at random from the lithic assemblage of Area C2, located in the middle of the settlement and one of the excavation loci in which several superposed Neolithic levels can possibly be distinguished (cf., 'upper–middle' and 'middle–lower' levels in Table 1) (Fedele 2008, fig. 3; Fedele 2009, figs 7 and 10).[3] However, no significant technological or typological difference between levels is apparent.

[3]Only the assemblage from Area C2, and two others from the western area of the site, have been systematically analysed up to now (Fedele and Zaccara 2005; and data on file). The collections housed at the National Museum in San'ā' are not presently accessible.

Table 1 *The list of Neolithic obsidian artefacts analysed for this study: the descriptions include linear measurements (mm) and weights (g)*

Artefact		*Description*
Study number	*Catalogue number (and excavation cut)*	
Debitage elements		
		Elements from upper-middle levels
1	0906 (E90)	Tertiary flake from single platform core (distal fragment). Platform and bulb are missing. The piece is strongly patinated. 21 × 18 × 2 mm. 1.0 g.
2	0909 (E90)	Tertiary flake from single platform core. Platform is linear and bulb is flat. The piece is strongly patinated. 20 × 17 × 5 mm. 1.7 g.
3	0914 (E90)	Chip. 0.2 g.
4	0931 (E91)	Secondary flake from 90° platform core. Platform is linear and bulb is diffuse. 9 × 22 × 5 mm. 2.1 g.
6	0970 (E93)	Chip. 0.1 g.
7	1011 (E93B)	Tertiary flake from single platform core (distal fragment). Platform is unfaceted, trapezoidal and bulb is unidentifiable. 20×10×3 mm. 0.6 g.
8	1087 (E94)	Tertiary flake from single platform core (distal fragment). Platform and bulb are missing. The piece is strongly patinated. 17 × 16 × 5 mm. 1.5 g.
9	1094 (E94)	Tertiary flake from opposed platform core (distal fragment). Platform and bulb are missing. 14 × 10 × 3 mm. 0.5 g.
11	1181 (E97A)	Tertiary flake from 90° platform core. Platform is unfaceted, triangular and bulb is diffuse. The piece is strongly patinated. 21 × 15 × 6 mm. 1.3 g.
12	1231 (E97C)	Secondary blade from single platform core (missing proximal end). Platform and bulb are missing. 35 × 11 × 3 mm. 1.3 g.
13	1233 (E97C)	Tertiary flake from single platform core (mesial fragment). Platform and bulb are missing. 11 × 16 × 3 mm. 0.8 g.
14	1249 (E98A)	Tertiary flake from single platform core (distal fragment). Platform and bulb are missing. 20 × 16 × 3 mm. 1.6 g.
15	1291 (E99C)	Tertiary flake from 90° platform core (missing platform). Platform and bulb are missing. The piece is lightly patinated. 14 × 15 × 6 mm. 1.5 g.
16	1293 (E99C)	Secondary flake from single platform core (missing distal end). Platform is crushed, irregular and bulb is flat. 23 × 14 × 5 mm. 1.7 g.
		Elements from middle-lower levels
17	1153 (E95A)	Tertiary flake from single platform core (missing distal end). Platform is crushed, trapezoidal and bulb is scaled. The piece is lightly patinated. 18 × 12 × 2 mm. 0.7 g.
18	1201 (E97B)	Reflex tertiary flake from single platform core. Platform is unfaceted, irregular and bulb is scaled. 22 × 17 × 2 mm. 0.9 g.
19	1354 (E99D)	Tertiary flake from 90° platform core (mesial fragment, missing left side). Platform and bulb are missing. 12 × 12 × 3 mm. 0.9 g.
20	1355 (E99D)	Tertiary flake from 90° platform core (distal fragment). Platform is unfaceted, irregularly triangular and bulb is flat. 17 × 16 × 3 mm. 0.7 g.
Retouched tools (all from upper levels)		
5	0967 (E93)	Triangle on tertiary flake from single platform core (fragment). Platform and bulb are missing. Retouch is inverse, bilateral, abrupt, stepped. The piece is strongly patinated. 15 × 17 × 5 mm. 1.1 g.
10	1135 (E95)	Backed element on tertiary bladelet from single platform core (mesial fragment). Platform and bulb are missing. Retouch is direct, right, partial, slightly invasive, abrupt, scaled. 15 × 10 × 3 mm. 0.5 g.

Figure 2 *Wādī ath-Thayyilah 3: the obsidian artefacts analysed for this study.*

Over 15 000 artefacts have been collected from WTH3 by hand-picking and sieving. General information obtained from the site collection, and the particular examination of the lithic artefacts from Area C2, show the presence of seven rock types among the raw materials: chert/flint, obsidian, metamorphic quartzite, quartzarenite, siliceous rocks of volcanic origin, basalt and granite (Fedele and Zaccara 2005, table B). The latter two were mainly used for macroliths (larger-sized tools; e.g., Fedele 2008, fig. 8 m) and other heavy-duty implements. Neolithic obsidian at WTH3 is represented by a dark black variety and is the second most exploited raw material after chert/flint, accounting for about 25% on average (e.g., 171 out of 816 artefacts, or 21%, in Area C2 east). It was brought to the site as both wadi cobbles and angular clasts ('chunks'); however, it is difficult to distinguish between these two kinds of supply in the analysed sample, because of insufficient preservation of cortex (Fig. 2). The existence of particular relationships between obsidian wadi cobbles used at the site and source selection, if any, awaits to be explored on the basis of a suitable sample.

The technological and morphological characteristics of the entire lithic collection from WTH3 have been outlined elsewhere (Fedele and Zaccara 2005, 226–39; Fedele 2008, 162–4). Suffice here to record, among them, a highly developed hafted-tool component, scant interest in formal blades, and—concurrently—expedient utilization of blanks. Toolkits are dominated by various kinds of scraping, boring and cutting implements, among which endscrapers, perforators, rabots and discoid core-tools display particular standardization. The tool to debitage ratio averages 20–40%, according to activity loci; and a 'tool' is here defined as including both fashioned and utilized pieces (Andrefsky 2005, 76). The industry as a whole is not microlithic. Like the tool rate, a tendency to small size appears to vary according to loci, with Area C2 corresponding in fact to one of the most 'microlithic' locations recognized at the site (Fedele and Zaccara 2005, fig. 17).

Almost all the artefacts selected for the present analysis belong to the debitage class, being mainly flakes (15), with the addition of one blade and two chips (working debris of small dimensions, <10 mm). The flakes mainly come from single-platform cores, followed by 90°-platform and opposed-platform cores. Several artefacts present strongly patinated surfaces. The small size of the sampled artefacts, ranging from 9 to 22 mm in flake length, is a result of convenience in sample selection. In fact, the obsidian component at WTH3 shows no more than a modest microlithic trend compared to the rest of the industry. Obsidian was not exploited very differently from chert or flint (Fedele and Zaccara 2005, 236), apart from hints of an economical use of the material available, as expressed in the amount of small debitage and small tools (for a similar observation, cf., Khalidi 2009, 284).

Two retouched tools were included in the sample, both microlithic (Fig. 3). One is a triangular element with inverse, bilateral, abrupt retouch, not obtained by the microburin technique but manufactured on a tertiary flake from a single-platform core. The second tool is a snapped (or fragmentary?) backed bladelet showing a direct, right, partial, slightly invasive, abrupt, scaled retouch: a rare artefact, considering that retouched backed pieces are less than 5% of tools, and backed bladelets much less. The snapping technique was reported as a major reduction strategy for the production of purposely shaped, small-sized—that is, microlithic *lato sensu*—elements in Neolithic highland Yemen, as particularly demonstrated at WTH3 (Fedele and Zaccara 2005, 238; Fedele 2008, 163–4; F. G. Fedele, on file, based on surface samples from eastern plateau sites). Intentional breakage may indeed approach 'a geometric conception' (e.g., Roubet and Lenoir 1997, 161, 175), but remains distinct from formal geometric microlith production (see also Neeley and Barton 1994).

To add perspective, a brief mention should be made of geometric microliths proper; that is, those resulting from standardized geometric production, whose appearance in Yemen would postdate the Neolithic. The production of geometric microliths is well documented in the Horn of Africa, where it could be connected with particular hunting and cultivation activities and has a longer tradition (Phillipson 1993, 85–6, 99–100). On the Tihamah coastal plain of southwestern

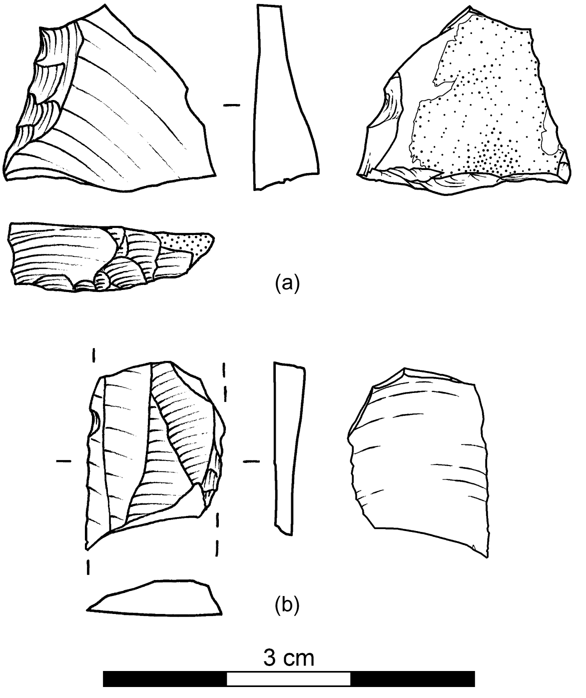

Figure 3 *Wādī ath-Thayyilah 3: the obsidian retouched tools numbers 5 and 10 (see Table 1)—(a) triangle; (b) backed bladelet (drawings by M. Pennacchioni).*

Arabia, however, their use is identified as a Bronze and Iron Age phenomenon, from the third millennium BC onward (Khalidi 2009, with references). This late appearance, as part of a bipolar-flaking package and in the context of increased, specialized obsidian exchange, can presumably be traced to an African origin (Khalidi 2009, 284). On the African side, obsidian geometric microliths notably occur at coeval sites in Djibuti (Asa Koma; Joussaume 1995, 32–6) and in the Agordat (Arkell 1954) and Adulis areas of Eritrea (Paribeni 1907; cf., Zarins 1989, 359, for dating).

ANALYTICAL METHODS

Geochemical analyses of each sample and find were carried out at the Department of Earth Sciences, University of Calabria, Italy, using a scanning electron microscope equipped with an EDS system (EDAX GENESIS 4000) to determine the major element composition and LA–ICP–MS to determine the trace element composition. The LA–ICP–MS equipment was an Elan DRCe (Perkin Elmer/SCIEX), connected to a New Wave UP213 solid-state Nd–YAG laser probe (213 nm). Samples were ablated by laser beam in a cell, and the vaporized material was then flushed (Gunther and Heinrich 1999) to the ICP, where it was quantified. The procedures for data acquisition were those normally used in the Mass Spectroscopy Laboratory of the Department of Earth Sciences, University of Calabria (Barca *et al*. 2007). In particular, for all analyses a transient signal of intensity versus time was obtained for each element using a 60 s measurement of background levels (acquisition of gas blanks) followed by 60 s of ablation and then

60 s of post-ablation at background levels. Data were transmitted to a PC and processed by the GLITTER program. The constant laser repetition rate was 10 Hz and the fluence about 20 J cm^{-2}. Each ablation crater was generally 50 μm in diameter and nearly invisible to the naked eye.

Only two point analyses were carried out on portions of archaeological fragments without roughnesses or alterations, and were sufficient to assign provenance. In order to remove any trace of soil, each find was cleaned by ultrasound in Millipore water. Calibration was performed on glass reference material SRM612–50 ppm by NIST (National Institute of Standards and Technology) in conjunction with internal standardization, applying SiO_2 concentrations (Fryer *et al*. 1995) from SEM–EDS analyses. In order to evaluate possible errors within each analytical sequence, determinations were also made on the SRM610–500 ppm by NIST and on BCR 2G by USGS glass reference materials as unknown samples, and element concentrations were compared with reference values from the literature (Pearce *et al.* 1997; Gao *et al*. 2002). Accuracy, as the relative difference from reference values, was always better than 10%, and most elements plotted in the range ±5%.

RESULTS

As a first phase, analyses were carried out on the five representative samples made available from the obsidian outcrops of the Jabal Isbīl and Jabal al-Lisī complexes in the Dhamār-Radā' volcanic field of Yemen (Chiesa *et al*. 1983). Four samples (Y431, Y452, Y475 and Y490) come from the obsidian lava flows of Jabal Isbīl, and sample Y475, in particular, had been collected near the village of Jarf Isbīl. A fifth sample, Y529, comes from Jabal al-Lisī. Subsequently, the 20 selected archaeological fragments of worked obsidian from site WTH3 were analysed. Tables 2 and 3 list, respectively, the major element composition, determined by SEM–EDS, and the composition of rare earth and other trace elements, determined by LA–ICP–MS, for both the geological samples and the archaeological specimens. Each major and trace element quantity in the tables represents the mean value of two analyses.

The major element concentrations show little differences among the geological samples studied. The three samples from the Jabal Isbīl volcano proper, Y431, Y452 and Y490, show concentrations of SiO_2 ranging from 72 wt% to 74 wt%. The other two samples, obtained, respectively, from Jabal al-Lisī (Y529) and Jabal Isbīl/'Jarf Isbīl' (Y475), show slightly higher SiO_2 concentrations (around 76.0 wt%); together with more variable, for all samples, concentrations of Al_2O_3 (8.25–14.6 wt%), FeO (1.5–8.5 wt%) and Na_2O (4.4–7 wt%), and very low concentrations of CaO (0.4–0.6 wt%), below the detection limit in some cases (Y490 and Y529). The concentrations of K_2O, at around 4 wt%, are essentially constant. The archaeological obsidian shows low variability in the major element concentrations: SiO_2, 74.4–76.6 wt%; Al_2O_3, 11–12 wt%; FeO, 2.3–4 wt%; CaO, 0.2–1 wt%; Na_2O, 4–7 wt%; K_2O, 3.6–4.8 wt%.

The LA–ICP–MS analyses allowed a complete geochemical characterization of the specimens in terms of trace (including rare earth) elements, highlighting differing chemical behaviour between the different samples. The elements showing the major differences between the samples are: Hf, Nb, Pb, Rb, Ta, Th, Y and Zr, and the majority of REE. In particular, the archaeological obsidian can be separated into two groups. The first (artefact group 1) is constituted by 14 specimens—2, 3, 4, 5, 6, 7, 8, 9, 10, 12, 16, 17, 18 and 20—giving the following compositional values: Hf, 22–35 ppm; Nb, 110–150 ppm; Pb, 27–44 ppm; Rb, 206–283 ppm; Ta, 7–11.5 ppm; Th, 28–42 ppm; Y, 103–161 ppm; Zr, 840–1352 ppm. The second group (artefact group 2) includes six specimens—1, 11, 13, 14, 15 and 19—with compositional values as follows: Hf,

Table 2 *The major chemical composition of obsidian by SEM–EDS: geological samples from the Dhamār-Radā‘ volcanic field, Yemen, and archaeological specimens from site WTH3, Wādī ath-Thayyilah, Yemen*

	SiO_2	Al_2O_3	*FeO*	*CaO*	Na_2O	K_2O
Geological samples from the Dhamar-Rada' volcanic field, Yemen						
Y431	72.0	8.3	8.3	0.38	7.0	4.1
Y452	74.0	14.6	1.5	0.56	5.2	4.2
Y475	76.2	11.3	3.2	0.57	4.4	4.4
Y490	73.4	10.2	5.3	n.d.	7.0	4.1
Y529	76.3	11.4	2.7	n.d.	5.3	4.3
Archaeological specimens from site WTH3, Wadi ath-Thayyilah, Yemen						
1	75.2	11.6	3.1	0.54	5.5	4.0
2	74.8	11.5	3.0	0.35	5.9	4.5
3	75.0	11.8	3.0	1.09	5.0	4.1
4	75.6	11.4	3.0	0.44	5.5	4.0
5	75.9	11.6	2.4	0.18	5.7	4.1
6	76.1	11.5	2.7	0.38	5.3	4.0
7	76.1	11.5	2.3	0.37	5.2	4.5
8	74.6	12.2	3.0	0.91	5.1	4.1
9	76.4	11.1	2.8	0.50	4.9	4.4
10	76.0	11.4	2.7	0.34	5.3	4.2
11	74.5	11.9	2.6	0.55	6.9	3.6
12	75.7	11.5	2.5	0.38	5.8	4.1
13	75.2	11.6	3.1	0.43	5.7	4.0
14	75.4	11.2	4.1	0.52	4.0	4.8
15	75.9	11.5	2.5	0.59	5.2	4.3
16	76.0	11.5	2.8	0.53	4.6	4.6
17	75.8	11.6	2.7	0.51	5.2	4.3
18	76.4	11.1	3.1	0.37	4.2	4.8
19	75.3	11.5	2.9	0.70	5.5	4.1
20	75.7	11.4	2.7	0.76	5.1	4.3

53–58 ppm; Nb, 221–248 ppm; Pb, 70–87 ppm; Rb, 461–542 ppm; Ta, 17–21 ppm; Th, 61–76 ppm; Y, 274–311 ppm; Zr, 1802–1990 ppm.

The trace elements Cr, Co, Ni, Cd and Sb were also determined, but their concentrations were often below the detection limits and, in general, they cannot help in provenance study .

The geological samples show large geochemical variability. In particular, sample Y452 presents concentrations that are very low as well as very different from the other samples; sample Y475 shows concentrations in the range of artefact group 1 for all trace elements; and samples Y490 and Y529 are characterized by trace element concentrations close to those of artefact group 2. Finally, geological sample Y431, which shows highly variable trace element concentrations, does not match with either group.

DISCUSSION

In order to assign provenance of each fragment, the geochemical results obtained from the geological and archaeological obsidian were compared using binary and ternary diagrams. In the SiO_2–Na_2O diagram (Fig. 4) both the geological and archaeological obsidian show a scattered distribution. The archaeological specimens form a large group overlapping the geological

Table 3 *Concentrations (in ppm) of the elements determined by LA–ICP–MS analysis in the geological and archaeological samples*

	Geological obsidian					*Archaeological obsidian*																			
	Y431	*Y452*	*Y475*	*Y490*	*Y529*	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*	*13*	*14*	*15*	*16*	*17*	*18*	*19*	*20*
Sc	3.58	5.08	—	1.26	3.21	4.90	3.98	2.38	1.25	2.81	1.14	2.68	2.64	3.46	—	1.84	1.88	—	3.23	3.02	2.63	4.1	—	2.53	1.83
Ti	3 023	713	939	1 447	814	962	1 172	1 116	972	907	904	1 319	1 410	1 139	1 086	933	1 224	1 023	814	909	1 185	1 159	1 181	839	1 130
Mn	2 151	452	478	1 148	304	325	410	393	396	428	400	472	515	423	408	319	410	341	381	366	394	428	399	355	441
Cu	7.0	2.7	2.4	10.3	3.3	1.8	2.7	2.5	3.4	3.6	5.2	4.9	1.7	4.7	4.7	2.7	2.1	5.7	4.3	3.6	1.9	3.4	1.7	1.9	3.0
Zn	388	113	180	453	302	338	252	230	227	214	258	180	190	285	183	322	223	421	355	357	184	202	196	365	220
As	4.95	3.42	6.43	13.35	11.41	11.2	3.13	2.05	8.25	13.9	2.18	4.31	3.72	3.62	—	—	—	13.98	5.6	6.01	3.67	—	—	2.5	3.95
Rb	247	151	266	364	539	482	283	257	250	244	237	227	206	283	273	461	265	477	542	520	229	281	277	521	269
Sr	2.58	3.72	1.50	4.83	1.14	0.18	0.42	1.80	—	0.24	2.59	1.14	1.72	1.54	1.89	—	0.64	0.64	0.10	0.22	0.33	0.82	4.75	0.12	0.32
Y	197	71	130	237	216	311	147	142	140	127	135	103	111	156	155	285	143	274	277	292	136	161	146	283	149
Zr	1 569	311	1 179	2 120	1 381	1 990	1 257	1 211	1 285	1 180	1 214	840	924	1 313	1 296	1 860	1 185	1 802	1 886	1 974	1 147	1 357	1 242	1 898	1 259
Nb	250	101	173	347	325	221	139	134	141	130	129	110	110	151	144	234	134	236	246	248	122	150	139	247	145
Mo	11.3	5.2	4.5	17.1	4.8	6.2	5.7	5.8	10.0	5.6	6.7	7.5	5.9	5.1	5.6	6.9	6.1	8.1	6.7	7.0	4.4	6.7	4.7	8.3	4.8
Sn	11.1	8.7	8.4	18.2	20.3	16.0	7.9	8.3	11.7	11.7	10.0	6.5	8.4	9.4	9.0	16.1	8.2	18.2	18.3	17.5	7.4	9.6	7.3	18.0	6.6
Ba	13.7	31.8	11.5	20.3	1.8	0.2	0.8	3.4	—	1.2	21.0	2.6	5.4	1.4	12.5	1.8	2.4	—	—	0.8	1.3	3.5	—	—	1.5
La	203	63	88	275	75	79	79	88	87	97	80	100	113	94	90	79	94	76	83	85	94	94	92	79	99
Ce	390	136	187	517	174	190	179	193	192	203	184	214	238	206	212	191	205	183	206	197	197	206	207	195	210
Pr	43.2	15.7	21.4	55.6	20.7	25.8	21.3	23.3	23.4	23.0	20.4	24.9	27.2	25.5	25.3	25.0	24.3	24.0	25.5	26.0	24.5	25.8	24.1	24.9	25.3
Nd	172	59	79	192	82	110	88	92	96	98	84	91	105	99	101	108	94	97	111	108	96	100	91	102	99
Sm	35.2	12.4	18.9	33.6	23.0	32.2	20.0	23.1	15.1	23.0	17.6	19.4	23.2	24.2	19.2	34.1	23.7	32.0	35.3	37.3	24.8	26.6	21.6	32.1	26.0
Eu	3.56	0.44	0.81	2.06	1.12	0.71	0.50	0.58	0.63	0.43	3.06	0.66	0.61	0.59	—	0.99	0.45	—	0.49	0.60	0.44	0.81	0.52	0.59	0.68
Gd	34.6	11.7	16.2	33.6	27.0	47.2	25.1	23.8	21.8	20.3	32.4	17.8	19.8	26.0	25.6	39.8	19.9	35.4	41.2	41.7	25.4	26.0	24.0	39.3	23.7
Tb	5.62	2.02	3.09	6.44	5.33	7.34	4.11	3.94	3.55	4.24	3.84	2.93	3.05	4.18	3.85	6.93	4.13	5.58	7.50	7.95	3.83	4.01	4.12	7.41	4.22
Dy	38.2	12.6	24.0	41.0	37.3	53.6	24.5	26.9	22.3	51.0	52.5	18.7	19.7	29.9	29.6	52.9	25.9	43.4	52.1	53.5	26.4	30.9	28.0	50.2	26.8
Ho	8.14	2.38	4.15	8.58	7.72	11.51	5.22	5.37	5.28	6.27	4.72	4.11	4.13	5.79	4.04	11.08	5.39	10.16	10.81	11.06	5.63	6.15	5.21	10.05	5.36
Er	24.1	8.6	16.1	26.3	23.5	32.3	15.6	15.3	15.4	17.1	15.6	11.0	10.7	16.5	17.8	30.8	14.9	28.4	33.4	34.6	16.2	19.0	18.3	32.7	16.4
Tm	3.40	1.20	2.61	3.85	3.30	5.21	2.57	2.34	2.83	2.87	3.17	1.95	1.68	2.50	2.23	4.57	2.27	4.09	4.90	4.88	2.49	2.79	2.45	4.83	2.45
Yb	24.3	8.4	12.4	26.5	23.1	29.7	16.5	15.2	14.1	13.4	15.9	10.8	11.1	15.7	17.7	29.5	15.1	28.9	32.6	32.7	14.5	18.8	13.3	31.4	17.4
Lu	3.24	1.24	1.82	3.73	3.16	4.13	2.37	2.25	2.89	1.94	3.62	1.65	1.73	2.40	2.83	4.76	2.44	3.97	4.45	4.53	2.60	1.97	2.31	4.70	2.49
Hf	40.9	11.2	29.8	52.2	42.3	52.6	29.1	29.9	34.1	22.0	26.7	21.6	22.5	34.7	32.0	53.6	31.5	55.6	57.5	57.8	31.3	32.8	26.3	57.7	32.0
Ta	17.5	7.8	12.3	26.5	27.3	19.5	9.7	9.9	8.7	7.3	10.5	7.5	7.4	11.0	11.5	19.7	9.0	16.7	19.2	20.4	9.9	10.8	10.2	21.3	10.3
Pb	34.8	26.0	30.2	51.7	61.6	80.6	37.1	39.7	40.1	37.0	34.4	31.6	27.1	44.1	36.3	70.5	36.1	76.2	86.2	86.9	35.7	40.2	39.2	82.8	37.9
Th	39.4	18.4	34.5	63.6	83.2	76.0	38.2	38.9	39.2	34.7	32.4	28.1	28.8	41.4	41.9	69.5	35.0	61.3	72.7	76.0	38.9	40.9	41.3	71.3	38.3
U	8.3	4.6	7.9	14.8	19.9	16.0	7.2	7.6	7.9	6.6	6.7	5.6	4.9	8.0	8.0	13.6	7.0	14.4	16.5	16.1	7.8	7.3	7.2	15.5	6.9

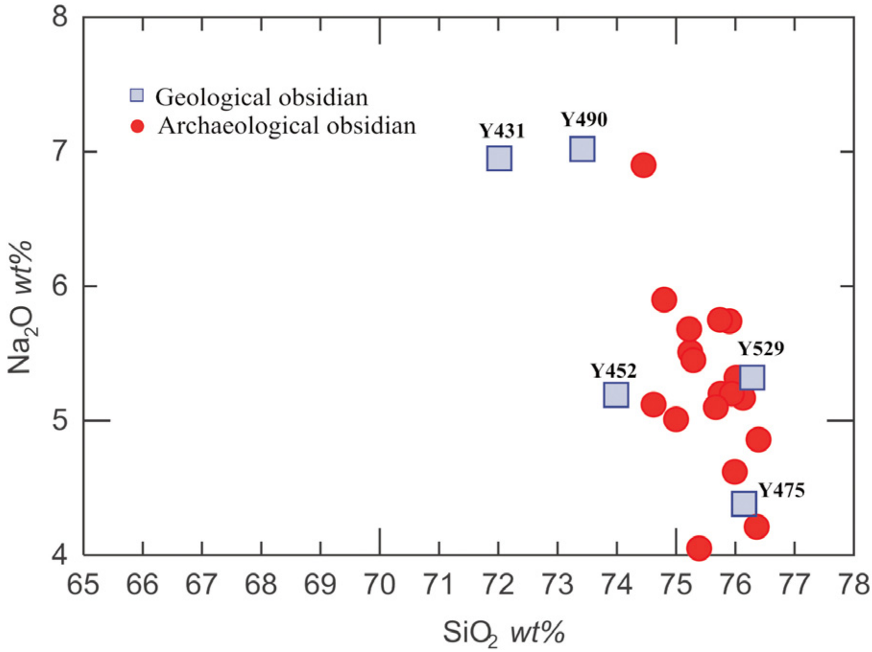

Figure 4 *The SiO_2–Na_2O diagram for the geological and archaeological obsidian analysed.*

samples Y452, Y475 and Y529; only specimen 11 plots near the geological samples Y490 and Y431. This kind of distribution cannot be connected with the two volcanic complexes analysed. From our analyses, it is clear that the obsidian types of Yemen cannot be discriminated by the major element concentrations alone, making the provenancing of archaeological samples impossible. On the other hand, the results of the LA–ICP–MS analyses were highly informative: they allowed discrimination among the geological obsidian and thus helped us to understand the provenance of the archaeological specimens. As a preliminary, however, a comparison was made concerning the geological obsidian between the data from this study and the published obsidian dataset from Yemen.

In the Rb–Ce diagram (Fig. 5 (a)) we compared our geological data with the source data of Francaviglia (1990b) and Khalidi *et al*. (2010). Sample Y529 corresponds well with the obsidian of the Jabal al-Lisī complex (Francaviglia 1990b), including the obsidian from Jabal al-Lisī proper and nearby Al-Gharga, which are geochemically very close to those of the Jirāb al-Souf source (Khalidi *et al*. 2010). Samples Y431 and Y490 plot in an area very near to the Jabal Isbīl obsidian. Sample Y475 from near the village of Jarf Isbīl, unfortunately not located more precisely during the sampling, matches the group 1 obsidian of Yafa' Ridge. Finally, sample Y452 plots very near the Hayd al-Halāl and Afar 1 groups.

In Figure 5 (b), the archaeological data and this comprehensive geological data set were compared in terms of the same Rb–Ce diagram. The 14 obsidian specimens in artefact group 1 plot precisely in the area of geological sample Y475 which, as mentioned above, is compositionally very similar to the Yafa' 1 group of Khalidi *et al*. (2010). The specimens in artefact group 2, on the other hand, plot near geological sample Y529, belonging to the Jabal al-Lisī complex. Two other diagrams, Y/Zr–La (Fig. 6) and Nd/Hf–Y/Zr (Fig. 7), confirm this double provenance

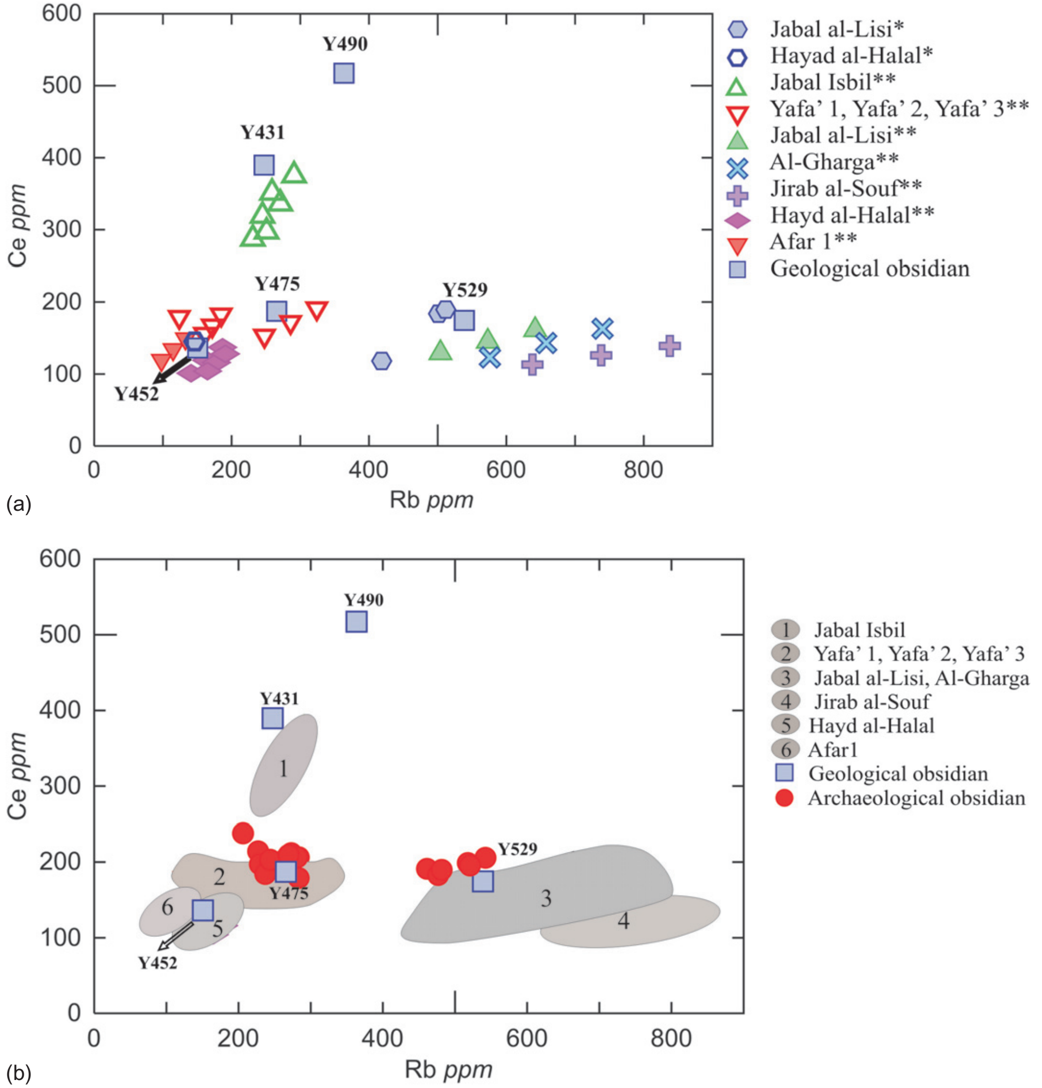

Figure 5 *(a) The Rb–Ce diagram for the geological obsidian analysed, compared with the data set in: *, Francaviglia (1990b); **, Khalidi* et al. *(2010). (b) The Rb–Ce diagram for the archaeological obsidian analysed, compared with the geological areas.*

for the analysed artefacts, which again plot in two distinct areas: artefact group 1 overlaps the geological obsidian of Yafa' Ridge and our Y475 sample; artefact group 2 plots close to geological sample Y529 and in proximity of the Jabal al-Lisī geological data sets of Francaviglia (1990b) and Khalidi *et al*. (2010).

Although minimal compositional heterogeneity was found in some cases (e.g., archaeological obsidians 5, 7, 8 and 18 in the Nd/Hf–Y/Zr plot), the observed variation was compatible with possible heterogeneity within the source, or the possible effect of weathering on the fragments.

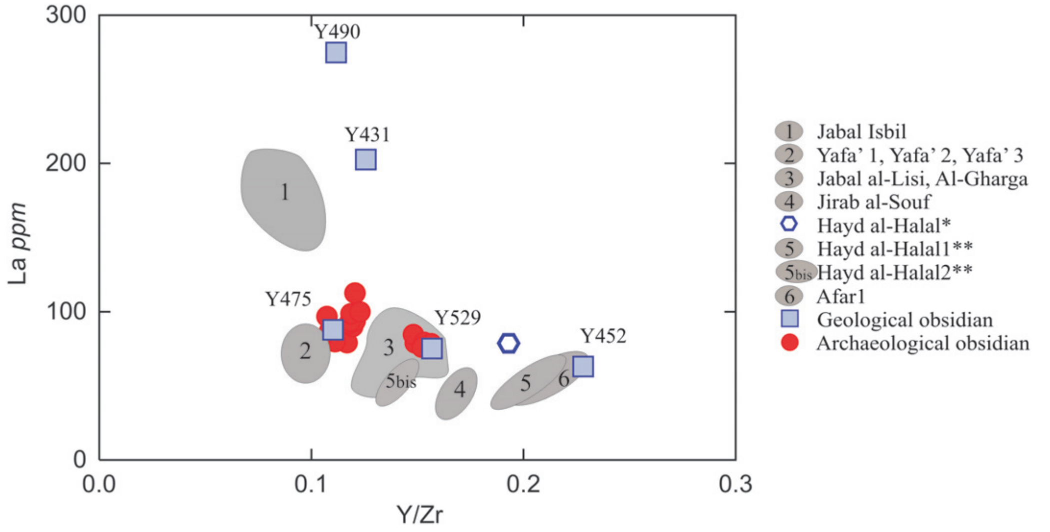

Figure 6 *The Y/Zr–La diagram for the archaeological obsidian analysed, compared with the geological areas: *, Francaviglia (1990b); **, Khalidi* et al. *(2010).*

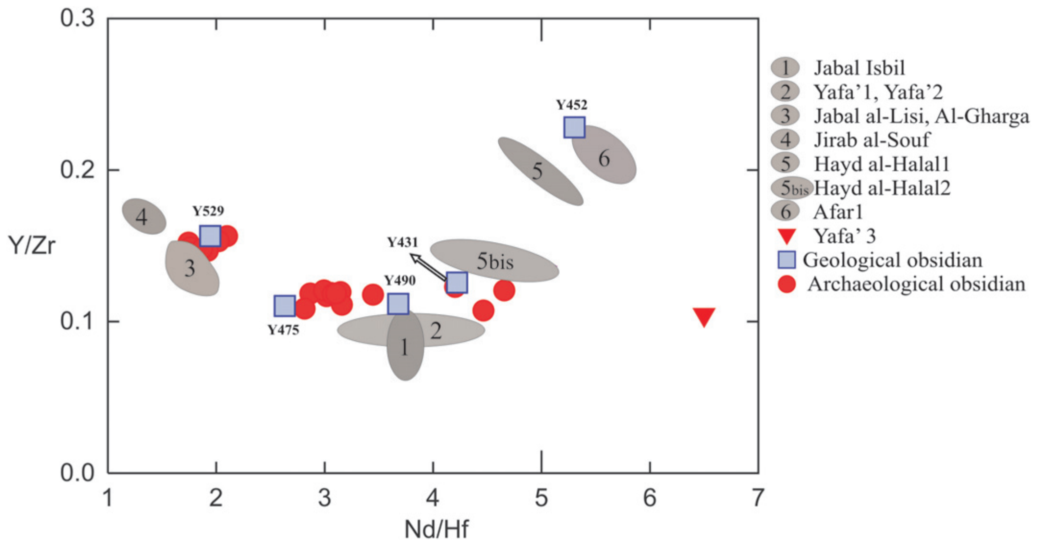

Figure 7 *The Nd/Hf–Y/Zr diagram for the archaeological obsidian analysed, compared with the geological areas.*

To resolve these issues, however, and particularly to account for the geochemical variability within sources, we need to expand our geological and geochemical knowledge of obsidian outcrops much further.

The Zr/Ce–Rb/Ce–Zr/Rb diagram (Fig. 8) once again confirms the good overlap both between artefact group 1 and the geological obsidian from Yafa' Ridge, and between artefact group 2 and the Jabal al-Lisī geological data set.

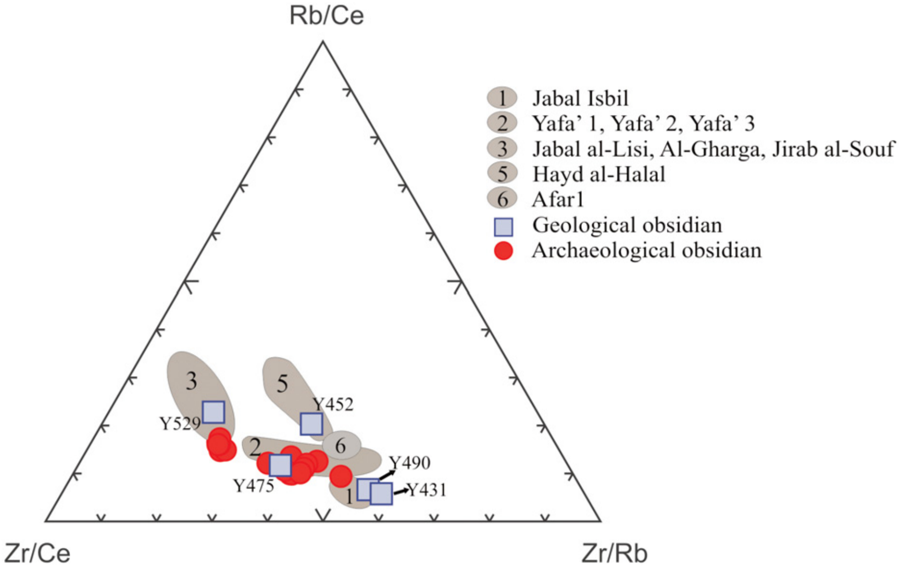

Figure 8 *The Zr/Ce–Rb/Ce–Zr/Rb triangular diagram for the archaeological obsidian analysed, compared with the geological areas.*

CONCLUSIONS

In this paper, we have endeavoured to obtain up-to-date, precise provenance determinations for a sample of 20 archaeological finds of Neolithic age (sixth to fourth millennium BC) from a well-excavated site on the eastern Yemen Plateau. This site, WTH3, can be used as a window on a still insufficiently studied period in a difficult-to-access region of highland Yemen. The size of the sample and its origin from a secure cultural context represent a valuable addition to the scanty, rather vaguely referenced Neolithic material examined by Francaviglia (1990b, 1996). The LA–ICP–MS system used in the present study produced precise analytical information on a number of trace elements, including rare earth elements. Comparison of the geochemical characteristics of the selected obsidian artefacts and those of geological raw materials allowed the provenance of each specimen to be established. In addition, particularly on the geological side, important matching information was derived from the obsidian data set now made available by the VAPOR project (Khalidi *et al*. 2010). The results reveal that the obsidian employed in manufacture came from lava flows in the Yemen central highlands, 70–75 km away to the SSW. In particular, a large majority of the artefacts studied (14 specimens) were made of obsidian from the Yafa' Ridge, and the other six of material coming from the Jabal al-Lisī volcanic complex.

The recent identification of the Yafa' Ridge as a major source of obsidian in prehistoric highland Yemen (Wilkinson *et al*. 1997, 122; Khalidi *et al*. 2010, 2336–7) resolved the issue of unknown provenance raised by Francaviglia in his pioneering work. The precise location of obsidian sources had remained an open problem in Yemen, especially because the conspicuous and supposedly choice outcrops of Jabal Isbīl could not possibly account for all the archaeological occurrences in the eastern Yemen Plateau and beyond (Francaviglia 1990b; see also Fedele 1990b; Fedele and Zaccara 2005, 229). However, the possibility that good-quality obsidian flows in Saudi Arabia or Yemen remain undetected is a problem to this day (cf., mentions in, e.g., Zarins *et al*. 1981, pl. 5C; Overstreet *et al*. 1988, 373–91; Zarins 1989, 346–58; Francaviglia 1990a, 46–8; Francaviglia 1990b, 133–4; Fedele and Zaccara 2005, 229; Khalidi 2009, 282).

Interpretations of our results in the perspective of obsidian exchange and highland intra-regional site interaction are clearly premature, and only interim considerations can be advanced. Overall, obsidian processing and use at Neolithic WTH3 appears to demonstrate steady access to tool-quality resources within an established central and eastern plateau network, and this already in a sixth to fourth millennium BC timeframe. Such a picture would contradict earlier views such as those of Wilkinson and Edens, when they predicted a pattern of localized and limited circulation from each highland source, with appreciable drop-off with distance (Wilkinson *et al*. 1997, 122). Obsidian quality may have played a distinct role in shaping highland procurement systems, as well as—perhaps—socio-economic interaction (cf., Khalidi 2009, 289).

However, the unknowns are overwhelming. To mention a few examples, in the Wādī Khamar Basin to the north-west of the Thayyilah–NAB district (Fedele 2009, 227–32) large-size obsidian finds observed at putative Neolithic sites could point to a different exchange network, if not a different source (F. G. Fedele, unpublished survey records 1987–90). In the Ramlat Sab'atayn desert to the east (Fig. 1, inset), well away from the known highland sources, obsidian frequency at a putative mid-Holocene site with bifacial lithic technology is a mere 3.3% (Di Mario *et al*. 1989, figs 1 and 2). Furthermore, to the exclusion of an intriguing hint provided by Pre-Neolithic WTH3 (see above), nothing seems to be known about obsidian exploitation in Yemen prior to the development of the Neolithic sociocultural systems.

In spite of its limitations, however, the information from WTH3 is significant on several counts. It contributes to the emerging picture of what may constitute the earliest obsidian procurement network in the mountainous part of Yemen, as outlined with admirable clarity by Khalidi *et al*. (2010). This adds in turn to our growing understanding of prehistoric highland resource acquisition, a subject that promises to be of increasing interest to researchers working across the broader Red Sea region, including northeastern Africa and extending to the Middle East. Finally, the new data have the potential to contribute to the definition of the upland Neolithic itself, a research area that is still dramatically under-explored.

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