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NON-DESTRUCTIVE ANALYSIS OF OBSIDIAN ARTEFACTS USING NUCLEAR TECHNIQUES: INVESTIGATION OF PROVENANCE OF NEAR EASTERN ARTEFACTS*

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A non-destructive analytical method using both instrumental neutron activation and proton-induced gamma ray emission techniques was developed to study the provenance of obsidian artefacts from Turkey, Syria and Iraq.

KEYWORDS: NEAR EAST, NEOLITHIC, OBSIDIAN ARTEFACTS, NON-DESTRUCTIVE ANALYSIS, FAST NEUTRON ACTIVATION TECHNIQUES, CORRESPONDENCE ANALYSIS

INTRODUCTION

The presence of obsidian artefacts at archaeological sites great distances from the known geological places of origin provides significant evidence of the movement of obsidian in ancient times. The determination of these geographical sources has always interested archaeologists for their relevance to ancient trade patterns. If the analysis of geological samples is easy providing the sample size is adequate, the analysis of ancient artefacts is more difficult because these objects cannot be always destroyed. The good results obtained in glass object analysis with a cyclotron fast neutron beam (Gratuze and Barrandon 1990) led us to use this, conjointly with proton-induced gamma ray emission, as a basis for a new non-destructive method allowing bulk analysis of obsidian artefacts. This method is used to assign source provenance to obsidian artefacts from Turkey, Syria and Iraq.

ANALYTICAL METHOD

For our work we used the experimental facilities of the Centre d'Etudes et de Recherche par Irradiation (CERI) of the CNRS in Orléans.

As obsidian is a natural glass, from an archaeological viewpoint, there are no problems regarding the technology. The determination of major elements is of less importance than for synthetic glass, and often the determination of trace elements is sufficient to relate artefacts

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to their sources. So instead of the two irradiations necessary for glass analysis (Gratuze and Barrandon 1990), a single irradiation followed by two direct gamma spectrometry measurements is carried out.

The obsidians are irradiated for four hours with a fast neutron flux produced by a deuteron beam impinging on a beryllium target (the energy of the deuterons is 17.5 MeV and the current beam intensity is 25 μA). A rotating device is used to obtain a homogeneous irradiation of large objects. The first count is ten hours following a cooling period of two to three days, the second count of ten hours is carried out after a cooling period of five to six days. Small discs of pure metallic element or salt pellets are used for standardization (Gratuze *et al.* 1992). These standards are irradiated simultaneously with sodium salt pellets, and a ratio ($K_{i/Na}$) between the specific activity of the radioisotope produced by the element *i* and the specific activity of ^{22}Na produced by the sodium is calculated. The following equation is used to calculate the results

$$\%i = \left(\frac{A_i}{A_{Na}}\right) * \frac{1}{K_{i/Na}}$$

with A_i and A_{Na} the activities of the radioactive nuclei produced by *i* and *Na* in the artefact.

Thirteen elements are then determined as a function of sodium concentration: Ca, Ti, Fe, As, Rb, Y, Zr, Nb, Sb, Cs, Ba, Ce, and U. Sodium is used as an internal standard to take into account the geometrical shape of the objects. The results are obtained in parts per million of the element for one percent of sodium.

In order to obtain absolute values, sodium is determined together with Li, F, and Al by particle-induced gamma ray emission (PIGE) using a 3 MeV proton beam from a Van de Graaff. The current beam intensity is in the range of 10 to 30 nA and the irradiation time is about 30 minutes. Reliable results could be obtained by PIGE analysis because obsidian is to some extent less affected by corrosion phenomena than glass. However, we can see in Table 4 that the sodium concentrations do not show great variations from one source to another. The ratios obtained by fast neutron activation analysis are therefore sufficient to determine the provenance of the artefacts.

Hence, 17 elements are determined using a non-destructive instrumental method. No interferences are observed for PIGE analysis and, among the 13 elements determined by fast neutron activation analysis, 12 are determined without interference or with negligible interference. However, for cerium a correction is made for a spectrometric interference due to uranium.

All the calculations are made with a program in a microcomputer (Apple Macintosh SE). Our results are given in ppm of the element for one percent of sodium, in percent of the element for Na and in percent of the oxide for Al.

The precision for the results obtained is in the order of 5 to 10% relative error. The detection limits for fast neutron activation analysis are in the range of 1 to 100 ppm. For PIGE analysis they are in the range of 0.01 to 0.1% for Na and Al and in the order of 30 ppm for Li and F.

In order to verify the accuracy of the analytical method and the precision of our results, some international standards of rocks were analysed: among them the SRM-278 obsidian rock from the National Institute of Standards and Technologies (NIST) (Glascock *et al.* 1988) (Figure 1). The difference between our values and the referenced values is around 5%

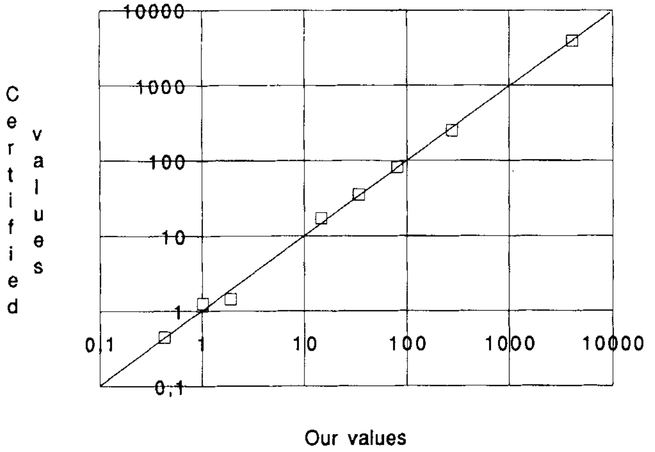

Figure 1 *Comparison of our values with the certified values of SRM-278 for U, Ce, Ba, Fe, Sb, Cs, Rb and Zr (all values in ppm/% Na).*

or less for Zr, Sb, Rb and Fe, around 10% for Ba and between 18 and 25% for Ce, Cs and U. The results obtained for Cs and U can be explained by the concentrations near the detection limits of our method for both elements.

A comparison was also made between our results and the results published by other authors on some geological sources (Figures 2 and 3). This comparison is supported by the results published by G. Schneider for the source at Kars 2 (Schneider 1990), by J. M. Blackman for the source at Sevan (Blackman 1981) and by V. Francaviglia and Y. Besnus for the source at Bingöl A (Francaviglia 1990; Cauvin *et al.* 1986). The relative difference between our values and these published values is in the range of 5 to 10% for most of the elements and does not exceed 25% except in the case of very low concentration values.

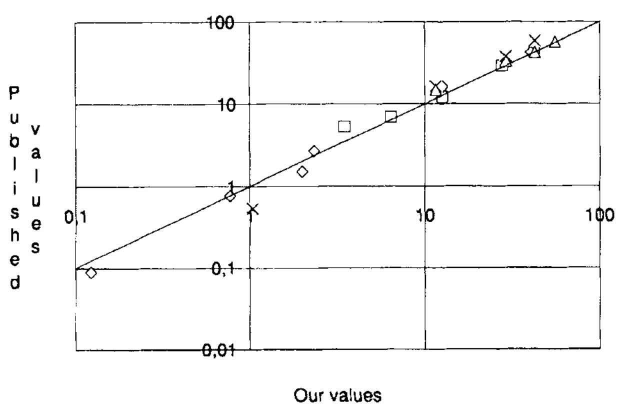

Figure 2 *Comparison of our results with the results published by other authors for Kars 2 (squares Ce, Rb, Y, Nb, Zr), Sevan (lozenges Ce, Sb, As, Rb, Cs, U) and Bingöl A (crosses for Y. Besnus (Ce, Ba, Y, Nb) and triangles for V. Francaviglia (Ce, Y, Rb, Nb)) (all values in ppm/% Na).*

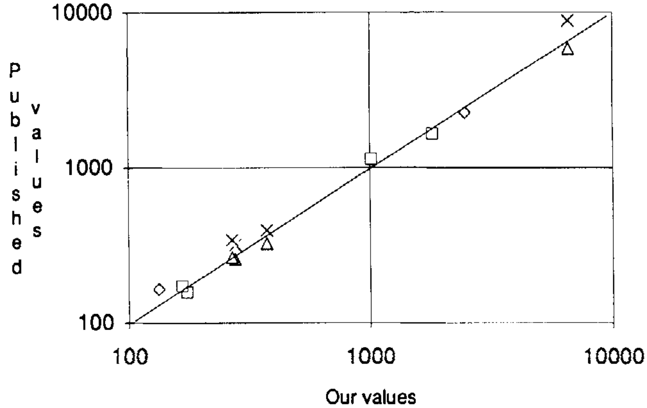

Figure 3 *Comparison of our results with the results published by other authors for Kars 2 (squares Ca, Ba, Ti, Fe), Sevan (lozenges Ba, Fe) and Bingöl A (crosses for Y. Besnus and triangles for V. Francaviglia (Fe, Ti, Ca, Zr)) (all values in ppm/% Na).*

RESULTS

The method was applied to the study of Near Eastern obsidian artefacts found on archaeological sites (Turkey, Syria and Iraq) dated from 8300 to 1300 BC. The obsidian sources in Turkey are located in four major zones: Cappadocia, Taurus, Lake Van and Armenia (Figure 4). A characterization of the sources situated in these regions was made.

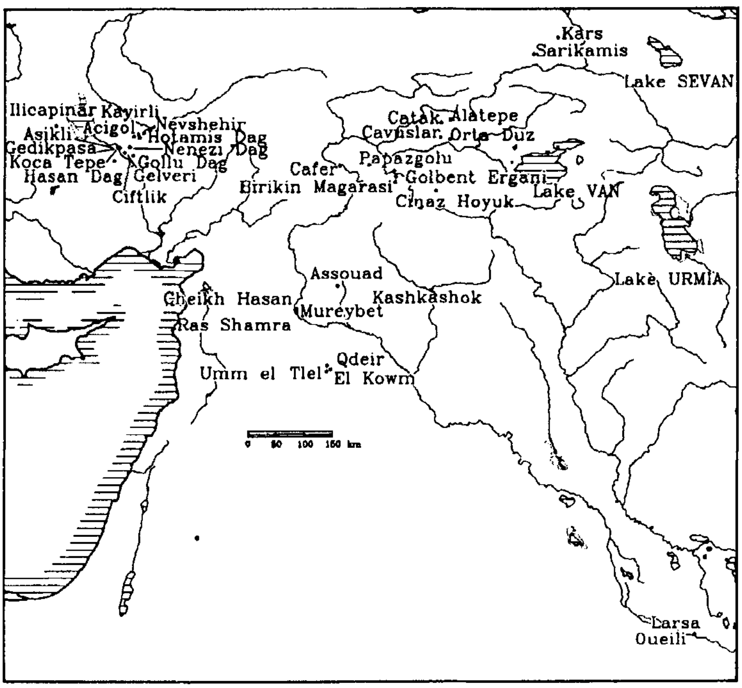

Figure 4 *Location of archaeological sites and geological sources.*

Table 1 *Chronology of the archaeological sites*

Archaeological sites	*Excavated by*	*Artefacts sent by*	*Periods and cultures (radiocarbon dates BC, uncalibrated)*	*No. of artefacts*
Mureybet: phase II	J. Cauvin	M. C. Cauvin	8300 to 8000	1
Mureybet: phase III A			PPNA 8000 to 7800	4
Mureybet: phase III B			PPNA 7800 to 7600	2
Mureybet: phase IV A			PPNB 7600 to 6800	3
Cheikh Hasan	J. Cauvin	M. C. Cauvin	PPNA 8000 to 7600	3
Asikli Höyük	U. Esin	N. Balkan	PPNB	14
Assouad	J. Cauvin	M. C. Cauvin	Late PPNB 6500 to 6000	5
Qdeir	D. Stordeur	M. C. Cauvin	Final PPNB 5800	25
Umm el Tlel 3, 4	M. Molist	M. C. Cauvin	Final PPNB 5800	8
Kashkashok	T. Matsutani	Y. Nishiaki	Halafian 5500	8
Golbent Ergani		N. Balkan	PPNB	2
Birikin Magarasi		N. Balkan	PPNB	2
Papazgölü		N. Balkan	PPNB	2
Cinaz Höyük		N. Balkan	PPNB	3
Ilicapinar Konya		N. Balkan	PPNB	3
Nevshehir Kogulalti		N. Balkan	PPNB	4
El Kowm 2: soundings PPNB	D. Stordeur	M. C. Cauvin	Final PPNB 5800	4
El Kowm 2: PNA			PNA 5500 to 5000	3
Cafer: upper level II	O. Aurenche and J. Cauvin	M. C. Cauvin	PPNB of Taurus 7000 to 6500	5
Cafer: upper level III				3
Cafer: lower level XI			PPNB of Taurus 7300 to 7000	2
Cafer: lower level XII				2
Cafer: lower level XII				2
Gelveri		N. Balkan	Unknown	5
Gedikpasa		N. Balkan	Unknown	2
Koca Tepe		N. Balkan	Unknown	2
Oueili	J. L. Huot	E. Coqueugniot	Ubaid	5
Larsa	J. L. Huot	E. Coqueugniot	Middle Bronze Age	1
Ras Shamra	M. Yon	E. Coqueugniot	Late Bronze Age	9

For the Taurus region an intensive investigation of the Bingöl area (Bingöl, Orta Duz, Cavuslar, Alatepe, Catak) was carried out. These sources are located at 20 to 40 km north-east of the town of Bingöl (Cauvin *et al.* 1986). Sources from the Lake Van area (Nemrut Dag) and the north-east (Armenia, Erevan, Kars, Sarikamis, Lake Sevan) were also analysed. In Cappadocia different sources were studied (Acigol (Göldag), Ciftlik, Kayirli, Kömürcü, Bözköy, Hotamis Dag (Karaçaören), Hasan Dag (Helvadere)).

Figure 4 shows the archaeological sites which were studied. Their chronologies and the number of artefacts analysed for each of them are given in Table 1 (Aurenche *et al.* 1981; Cauvin 1987; Cauvin 1991). Obsidian artefacts found in archaeological deposits come from different regions. For Syria the sites are in upper Mesopotamia or the 'Jezirah' (Mureybet, Cheikh Hasan, Assouad, Kashkashok), in the oasis of El Kowm in the semi-desert steppe (Qdeir, Umm el Tlel, El Kowm 2) and on the coast (Ras Shamra). In Turkey the samples come from the eastern Taurus (Cafer Höyük, Golbent Ergani, Cinaz Höyük, Birikin Magarasi) or from Cappadocia (Asikli Höyük, Nevshehir Kogulalti, Ilicapinar, Gelveri, Koca Tepe, Gedikpasa) and in Iraq from lower Mesopotamia (Oueili, Larsa).

These different sites are spread in date between 8300 and 1300 BC (uncalibrated). The most ancient, those before 7500, represent the transition between hunting/gathering and agriculture at the beginning of the Neolithic (Mureybet phase II), and then the beginning of an agricultural economy (Mureybet phase III, Cheikh Hasan). Others belong to the later pre-ceramic Neolithic, the PPNB, between 7300 and 6000 BC; these are Mureybet phase IV, Assouad, all the sites of the eastern Taurus and, finally, Asikli in Cappadocia. The deposits of the site of El Kowm belong to a very last phase, appropriate for the desert, of the PPNB (between 6000 and 5500 BC), contemporary with the first ceramics in other regions; one of them (El Kowm 2, PNA) belongs to an even later phase with ceramics at the end of the sixth millennium. It is also at the end of the Neolithic that we have 'Halafian' (Kashkashok) and 'Obeidian' (Oueili) samples. Finally, the material from Ras Shamra and Larsa was recovered in an urban context of the Bronze Age.

In all the sites these obsidian objects imported from far away are relatively exceptional in relation to the rest of the artefacts recovered. Only in the villages of the Taurus and Cappadocia did this material form a substantial part of their lithic tools.

A comparison of our analytical method with previous work shows that, with the exception of strontium, scandium and manganese, we determine most of the elements used in the literature for source discrimination and that, even if we obtain our results in the form of ratio of concentration, a comparison is possible with other published data.

The Ba/Zr ratio was the primary discrimination technique (Renfrew and Cann 1964), but other ratios such as Nb/Y (Renfrew and Cann 1964), Na/Mn (Wright 1969) and Rb/Sr (Gale 1981) were used. In this work, we use a multivariate data processing method, correspondence analysis (Fenelon 1981; Rauret *et al.* 1987; Baxter 1989): this method allows the projection of the objects from the n-dimensional original space on to a two- or three-dimensional space whose axes are the linear combinations of the n original factors. During this process, the maximum amount of variance is retained. Among the 17 analysed elements, eight were retained for data processing: Ca, Ti, Fe, Y, Zr, Nb, Ba and Ce.

Seven different compositional groups are obtained for the artefacts, whose distributions are given in Table 2 (the first group can be divided into two subgroups, Gla and Glb, if we consider mainly the iron and titanium concentrations). The groups obtained with the sources are given in Table 3. The average chemical compositions of the groups are given in Table 4.

A comparison of these results shows that the first, the second, the third and the seventh groups of artefacts could be assigned respectively to our sources Bingöl 'A' or Nemrut Dag, Bingöl 'B', Kayirli and Hotamis Dag. At this time, we are still not able to distinguish between the Nemrut Dag and the Bingöl 'A' sources.

More results are available for the Nemrut Dag sources than for those from Bingöl. Works published by J. M. Blackman (1981) and V. M. Francaviglia (1990) show that different obsidian flows of quite similar composition, differing mainly in their iron composition, are identified in the Nemrut Dag region. The comparison with Blackman's work is only supported by five elements (Ce, Ba, Fe, Rb and Cs) whereas nine elements are used for Francaviglia's results (Ce, Ba, Fe, Rb, Ti, Y, Zr, Ca and Nb).

The compositions of our two subgroups, Gla and Glb, are not very different to some of these authors' groups (Table 4). But, if at one archaeological site we find the artefacts have the two compositions of the Bingöl area, we may suppose that the artefacts come from Bingöl, whereas if only the Bingöl 'A' composition is found, both solutions (Nemrut Dag and Bingöl) should be retained as we find archaeological sites with only the Bingöl 'B'

Table 2 *Compositional groups for the artefacts*

Compositional groups	*Archaeological sites*	*No. of artefacts*
1a	Assouad	2
	Kashkashok	1
	Cafer: upper levels	1
	Larsa	1
	Oueili	2
1b	Papazgölü	1
	Kashkashok	3
	Mureybet IV	1
	Golbent Ergani	1
	Cinaz Höyük	1
	Qdeir	5
	Cafer: upper levels	1
	El Kowm 2: sounding PPNB	2
	El Kowm 2: PNA	1
2	Mureybet III B	1
	Golbent Ergani	1
	Cinaz Höyük	2
	Papazgölü	1
	Birikin Magarasi	2
	Umm el Tlel	5
	Cheikh Hasan	1
	Assouad	3
	Qdeir	11
	El Kowm 2: PNA	1
	Cafer: lower levels	6
	Cafer: upper levels	6
	Kashkashok	4
3	Mureybet II, III, IV	8
	Cheikh Hasan	1
	Asikli Höyük	12
	Nevshehir Kogulalti	4
	Ilicapinar	3
	Gelveri	1
	El Kowm 2: sounding PPNB	1
	El Kowm 2: PNA	1
	Qdeir	9
	Ras Shamra	6
4	Cheikh Hasan	1
	Umm el Tlel	3
5	Ras Shamra	3
	Gelveri	4
	Koca Tepe	2
	Gedikpasa	2
	Asikli Höyük	2
6	Oueili	3
7	El Kowm 2: sounding PPNB	1

Table 3 *Geological source groups*

Regions	*Compositional groups*	*Geological sources*	*No. of analyses*
Taurus	Bingöl 'A'	Bingöl 1, 7, 9, 10	18
		Cavuslar	2
		Orta Duz	2
	Bingöl 'B'	Bingöl 3	4
		Alatepe	4
		Catak	4
Lake Van	Nemrut Dag	Nemrut Dag	7
Armenia	Sevan	Sevan	6
		Erevan	1
	Kars 1	Kars?	2
		Sarikamis	1
	Kars 2	Kars?	2
	Armenia	Armenia	1
Cappadocia	Acigol	Acigol	8
		Göldag	1
	Hotamis Dag	Karaçaören	1
	Hasan Dag	Helvadere	1
	Kayirli	Kayirli	2
	Ciftlik	Ciftlik	1
		Kömürcü	1
		Bözköy	1

composition. New investigations in both regions should be undertaken in the future to answer these questions and to try to distinguish more clearly between both sources.

For group 5, we may notice a similarity with the Cappadocian source identified as Nenezi Dag by J. Blackman (1986) and I. Perlman (Perlman and Yellin 1980). The provenances of artefacts of groups 4 and 6 are still not identified, but new sources from Lake Van, Taurus, Armenia, Cappadocia and Iran will be investigated in the near future to solve this problem and to confirm the provenance of group 5.

CONCLUSION

We may say then that in the Jezirah, where previous analyses (Cauvin 1991), including those of Mureybet II–III A, have shown that the obsidian came exclusively from Cappadocia until about 7800 BC, that after this date (Mureybet III B and Cheikh Hasan) it came from Cappadocia as well as from the regions of Bingöl or Nemrut Dag. These new contacts between the middle Euphrates and eastern Anatolia confirm similar conclusions reached from other data (Cauvin 1987).

Table 4 *Average chemical composition for source and artefact groups*

	Na (%)	Al_2O_3 (%)	Ce/Na ppm	Ba/Na ppm	Fe/Na ppm	Ca/Na ppm	Ti/Na ppm	Y/Na ppm	Zr/Na ppm	Nb/Na ppm	No. of analyses
Bingöl 'A'	4.3±0.2	11±0.5	42±6	1±1	6589±378	374±40	276±21	29±2	268±13	12±1	22
Bingöl 'B'	3.7±0.2	14.7±0.6	16±2	106±10	3433±178	1435±88	322±18	7±1	80±2	4.2±0.2	12
Nemrut Dag	4.2±0.2	11±0.3	41±0.5	n.d.	6150±275	514±138	295±9	28±2	267±20	14.5±0.5	2
Nemrut Dag			37	n.d.	5256	494	276	26	232	12.2	2
Nemrut Dag			42	n.d.	7139	340	307	28	268	14.3	1
Nemrut Dag			41	n.d.	8794	532	407	29	252	13.5	2
Sevan	3.5±0.2	14.7±0.2	12.7±1	134±16	2475±208	1939±177	285±15	4.6±2.5	44±2	9.4±0.6	7
Kars 1	3.3±0.2	14±0.3	15±1	100±13	2382±96	1095±75	259±12	8±1	45±3	4.5±0.2	3
Kars 2	3.3±0.2	13.8±0.2	13±0.5	175±18	1825±57	1014±57	167±1	6.4±0.3	28±1	3.5±0.2	2
Armenia	3.3±0.2	13.5±0.3	9	79	1284	1174	160	11	24	9	1
Acigol	3.3±0.2	12.9±0.3	7.3±0.7	1.3±1	1893±148	774±74	47±6	10±1	24±2	9±1	9
Kayirli	3.1±0.2	13.1±0.3	12±0.1	37±2	1700±30	994±60	107±0.1	7±1	22±1	7±1	2
Ciftlik			14±1	50±23	3130±350	985±96	123±14	10±1	25±2	7.3±0.4	3
Hotamis Dag	3.3±0.2	14.1±0.2	12	114	2807	1857	161	5.8	42	4.8	1
Hasan Dag	3.6±0.2	14.3±0.2	12	371	1799	1583	222	4.3	21.7	4.1	1
Nemrut I (Blackman)	3.8		49.4±0.2	27±6	5332±29						8
Nemrut II (Blackman)	4.2		49.8±3	29±4	6963±751						5
Bingöl (Francaviglia)	4.4±0.1	11.4±0.9	42.3		5866	325	259	33	266	14.8	9
Nemrut I (Francaviglia)	4.3±0.2	12.2±0.1	45.6	1.6	5177	549	279	31	258	17.9	12
Nemrut II (Francaviglia)	4.36±0.4	10.8±0.2	49.1		7358	475	371	33.9	282	19.7	3
Nemrut III (Francaviglia)	4.9±0.2	9.48±0.1	46.3		8656	451	428	33.2	267	18.2	33
Nenezi Dag (Blackman)	3.2±0.2		21±2	207±12	2608±140						2
Nenezi Dag (Perlman)	3.21			171	2754						18
Group 1	4.1±0.3	11.3±0.4	43.8±4	0.7±1.8	6315±937	429±119	283±42	27.8±4.4	279±21	12.6±1.6	
G1a	3.8±0.3	11.6±0.4	45.9±2	2±2.8	5190±187	423±138	233±12	30.9±4.1	295±10	14.1±1.2	7
G1b	4.2±0.3	11.2±0.4	42.5±4	n.d.	6990±305	432±114	313±16	26±3.7	269±20	11.7±1.2	16
Group 2	3.6±0.2	14.4±0.4	16.6±1.5	120±15	3630±252	1561±225	333±20	5.9±2	83.6±3	4.4±0.8	44
Group 3	3.1±0.2	13.2±0.5	11.5±1	50.7±14	1987±207	969±127	111±12	5.2±2	22.9±2	6.3±1	46
Group 4	3.6±0.1	14.6±0.4	15±2	70±6	3573±146	1017±94	269±18	6.6±1	68±3	4±0.5	4
Group 5	3.2±0.2	14.3±0.3	16.4±1.5	170±12	2474±134	2251±109	221±7	4.6±2	37.3±1	5.2±0.2	13
Group 6	3.2±0.2	14.2±0.4	10±0.2	11.5±1.3	1090±136	1309±97	116±2	7.9±1.6	20±0.6	15.2±0.6	3
Group 7	3.3	13.9	14.7	118	2404	1888	161	7.1	34	5.7	1

n.d.: not determined.

The archaeological importance of the source 'B' at Bingöl seems to enlarge as analysis goes on. We know through Besnus that the obsidian from the higher levels of Cafer Höyük comes from this source; this is true also for the lower levels of this site as well as for other prehistoric sites in the Taurus. Moreover, this obsidian is found abundantly in all periods on the Syrian sites of the Jezirah (Assouad, Kashkashok) and the semi-desert steppe (oasis of El Kowm). It remains meanwhile to distinguish the source 'A' more clearly in relation to the obsidian of Nemrut Dag which has a very similar composition.

As for the obsidian of Cappadocia, it constitutes in two forms (strata of Kayirli and of Nenezi Dag) the totality of the material used at Asikli, a village of the same region. It is even found much farther, on the archaeological site of Ras Shamra in the historical period. Towards the east it reached the oasis of El Kowm from the late PPNB.

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