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A REVIEW OF ARCHAEOMETRIC RESULTS ON SARAKENOS CAVE, GREECE: FIRST STABLE ISOTOPE DATA (^{18}O AND ^{13}C) ON MOLLUSK SHELL (*UNIO SP*) INCLUDING OSL DATING AND CHARACTERIZATION-PROVENANCE OF CERAMICS BY PXRF

Liritzis, I.[1,2,3], Drivaliari, A.[1] and Vafiadou, A.[1]

[1]University of the Aegean, Laboratory of Archaeometry, and, Laboratory of Environmental Archaeology & Preventive Conservation, 1 Demokratias Str., Rhodes, Greece
[2]University of Henan, Key Research Institute of Yellow River Civilization and Sustainable Development, School of Geography & College of Environment and Planning, Kaifeng, Henan, China
[3]The University of Edinburgh, School of History, Classics and Archaeology, College of Arts, Humanities & Social Sciences, Dept. of Archaeology, 4 Teviot Pl, Edinburgh EH8 9AG, Scotland

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corresponding author: I. Liritzis
(liritzis@rhodes.aegean.gr; ioliritzis@gmail.com)

ABSTRACT

Archaeometric analysis of human and environmental remains from Sarakenos cave (Kopais basin, Beotia, central Greece), include, ^{14}C dates of charcoal, optical stimulated luminescence (OSL) dating of early hearth, characterization of ceramics by XRF from Neolithic to Bronze age levels, and, the first stable isotope data (^{18}O and ^{13}C) of freshwater mollusk shells (*Unio sp*.). The excellent stratigraphical data in the cave has offered a succession of distinct cultural phases, dated from the Middle Paleolithic to the Middle Helladic times. The isotopic data have provided significant variations, indicating paleoclimatic fluctuations in (pen)-insular central Aegean in prehistoric times with broad but distinctive climatic phases, the OSL age reinforces Mesolithic period, while XRF and clustering implies Aegean-Mainland interaction and trade.

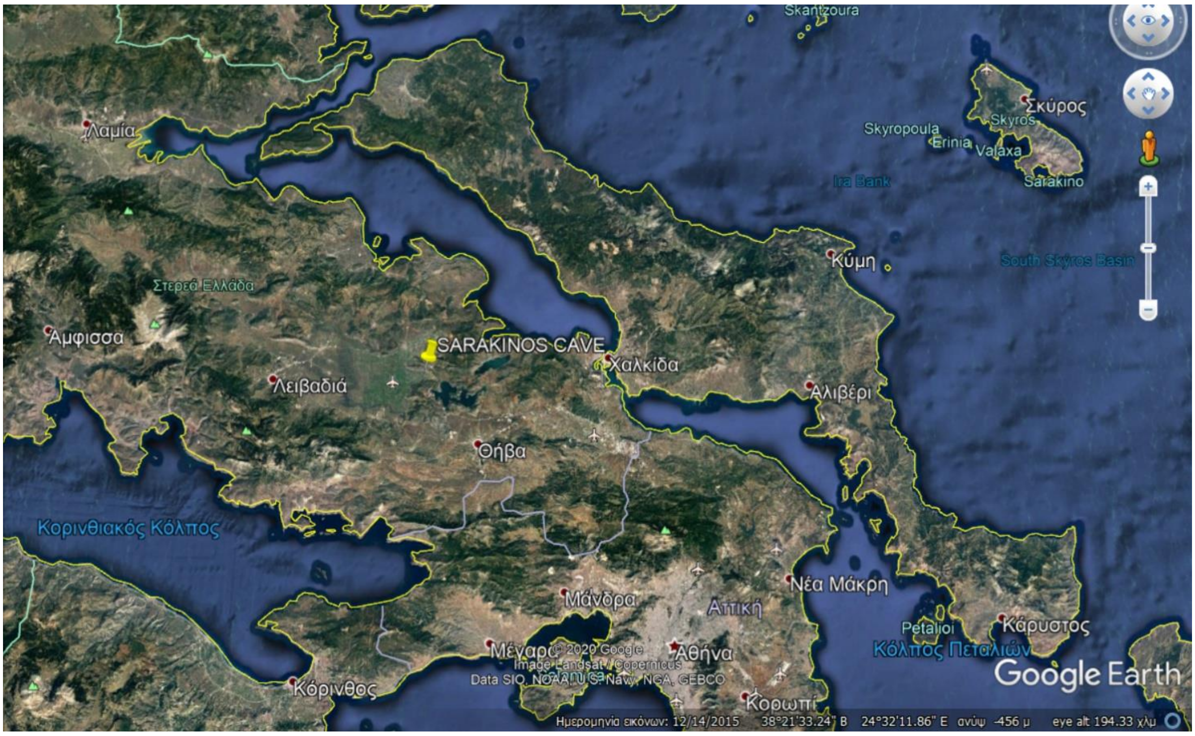
KEYWORDS: Palaeoclimate, neolithic, mesolithic, archaeology, XRF, luminescence, cave, Greece

1. INTRODUCTION

The cave of Sarakenos is situated in Boeotia, central Greece (Fig.1 Aegaen Sea by NASA, site no 2), overlooking the Kopais basin, a former a lake, as result of tectonic subsistence during the Pliocene and Pleistocene of the limestone bedrock (Papadopoulou-Vrynioti, 1990). This is one of the largest and most important cave of the area, which is known for the abundance of caves and rock shelters, situated within low calcareous formations of Jurassic or Cretaceous age and located at low levels, at the level of the former lake of Kopais; some of them functioned as swallow-holes, channelling the water of the lake to other lower basins or the sea.

The systematic excavation of the Sarakenos Cave in the former Kopais lake region (central Greece) (Fig.1) revealed Upper Palaeolithic to Middle Helladic (especially Mesolithic, Neolithic and Bronze Age) occupation remains on preserved deposits of 4 to 5m thick (Fig.2) (Sampson, 2008).

(A)



(B)

Figure 1. A. the cave, B. the location in central Greece

The stratigraphical data sequence in the cave indicates distinct cultural phases, from modern top surface to cave's natural bedrock. Exact dating of the strata due to absolute dating methods fixed the end of the Upper Palaeolithic by an age of about 12000 BC and the upper part of the stratum was dated to the beginning of the Mesolithic Age (*ca.* 9000 – 8500 BC) (see relevant articles in Sampson, 1998, 2000, 2008). The stratigraphy of three trenches and their dating (typologically but securely by ^{14}C) and the locations of sampling for shells and OSL dating unfolds in the following sections.

The aim of this work is a critical review on three issues, ceramic characterization and possible trade interactions from Neolithic to Bronze age periods, a detailed luminescence dating of a burnt soil from a hearth, and presentation of new data of stable oxygen and carbon variations (*ca.*2000-6000 BC) from freshwater mollusk shells (*Unio sp*.) in the cave, accompanied by a paleoclimatic discussion.

2. LAKE KOPAIS AND PALAEOCLIMATICS

Because of its importance as a natural karstic basin, Kopais has been the subject of extensive palaeoenvironmental studies since the 1970s. Greig and Turner (1974) published detailed pollen diagrams and Allen (1997) offered information on the vegetation from the Late Upper Palaeolithic on wards from two new cores.

Kopais is a natural basin in the northeast part of Boeotia created by tectonic activity some 10 million years ago. The area around the basin is a highly karstic landscape. Until the recent past, a big lake, differentiated from time to time by size, was covering the actual plain. During the Late Upper Paleolithic the Kopais had a vegetation typical of an open steppe and a dry and cold climate (*Artemisia, Graminae and Chenopods*). The Pleistocene- Holocene transition is recorded in the diagrams with the forest expansion (*Quercus, Juniperus, Pistacia, Ephedra*). At layers that correspond to 4000-3000 BC Quercus drops, possibly due to deforestation.

Analysis of the grain of the coring samples suggests that there was fluctuation in the lake levels during the Late Pleistocene and Early Holocene. There are also indications that the lake level dropped after 4000 and until 2500 BC. The first drainage works are possible to have taken place in the early 2nd millennium BC, under the aim to convert a big area of the lake into arable land.

The last layer above the bedrock yielded lithic finds and animal bones that are dated to the Upper Palaeolithic (Aurignacian period).

Soil and charcoal samples as well as charred seeds from the cave offered us information about the palaeoenvironment in the Kopais basin from the Palaeolithic to the Middle Bronze Age. The palynological assemblages that have been studied from the Sarakenos deposit show presence of Pine and Quercus and an increase of Leguminosae during the transition from Late Neolithic I to LN II (second half of the 5th mill. BC). The same species are present with small fluctuations during the Late Neolithic II and the EH II. In general, from the second half of the 5th mill. and until the 2nd millennium BC the plant species recorded in the cave pollen diagrams show the clear impact of the humans on the environment of the Kopais basin.

The formation of the lake was the outcome of tectonic movement that took place during the Pliocene and Pleistocene eras, and the dissolution of the calcareous rock caused by underground waters. From a geological point of view, the most prevalent rock is limestone, dating from the Mesozoic era. In the past, the water from the lake would be canalized towards the sea by means of an underground network of canals through the calcareous rock. Due to the progress of sedimentation, the lake was becoming very shallow and dry during the summer season, conveying a sense of seasonal differentiation between a lake and a swamp.

In 1983, two new cores were made that offered information on the vegetation from the Late Upper Paleolithic onwards. There are also indications that the lake level dropped between 4000 BC and 2500 BC (the dryness recalls similar widespread event at Egypt).

A sterile layer above contains only microfauna showing a long period of abandonment of the site, while it contains a huge concentration of ash and burnings. The presence of man at some chronological moment is certain, nevertheless without the presence of any specific finds.

The layers of burnt material are evident of man's entrance into the cave towards the end of the Palaeolithic or the beginning of the Mesolithic Age, as proven by three calibrated ^{14}C dates provided by charcoal samples from this stratum (DEM-1206, 9233+/-30 BP or 8530-8340 BC; DEM-1209, 9177+/-31 BP or 8450-8290 BC; and DEM-1210, 9230+/-30 BP or 8530-9340 BC). The first and third examples come from the same hearth, with a characteristic absolute precision. The second example comes from another hearth and is slightly more recent. These ages are contemporaneous to the earliest Mesolithic phase of the Cyclops Cave (Sampson, 1998).

A sample of the burnt material from the same hearth dated with the method of Optical Thermoluminescence offered an analogous age of 10110+/-750 BP (Liritzis, 2008).

3. THE STRATIGRAPHY, SAMPLING AND DATING OF CULTURAL LAYERS

From the Early Neolithic to the end of the period the occupation in the cave seems to be uninterrupted. All this long Neolithic sequence was explored mainly in trenches A and B (Figs 2, 3). A great number of macro and microfauna, particularly shells (*Unio sp.*) originate from all these strata (Wilczyński et al., 2016). Fresh water shells have been sampled within selected layers among the three excavations sections (trenches) and corresponding squares (rectangles), as indicated in Table 1. The placing of samples in the isotopic data plots follows the archaeological age (and absolute dating where available), as well as, considering the corresponding layer's depth within the three (3) trenches (A, B, and C).

Figure 2. Sarakenos Cave. Stratigraphical section, section B.

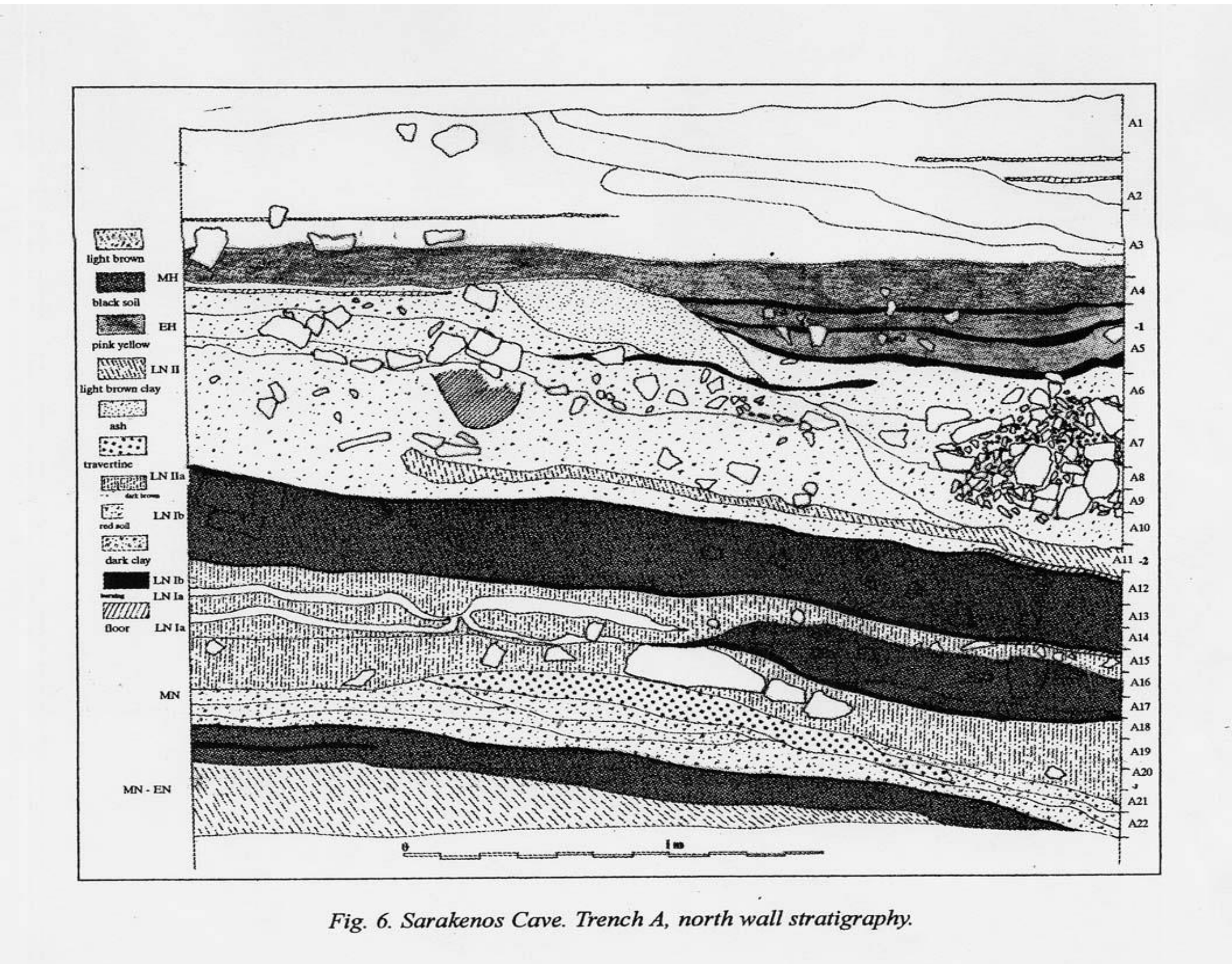


Figure 3. Sarakenos Cave. Stratigaphical section, Trench A, north wall stratigraphy (from Sampson et al., 2009, Fig.5).

A detailed description of stratigraphy and cultural occupation within the three (3) trenches of excavation is given by specialists in edited volume (Sampson, 2008) and in Sampson (1998, 2000).

Most freshwater mussel shells (Unio sp.)[1] have been identified among the faunal remains were collected from Trench A. (NB: Layer 7 at a depth of 1.40 m, Layer 8 at a depth of 1.60m dated between 4330 and 4250 BC, to Layer 20 from a depth of 2.90 to 3.00m of Middle Neolithic/Early Neolithic pottery occurred).

Figure 4. Characteristic bivalve Unio sp. Shell found in the cave. The faunal record in Sarakenos cave has been studied that includes mussels of shells Unio sp. (see, Wilczyński et al., 2016).

The palaeolithic layer in Trench B (Fig. 2) is thin and only covers the bedrock, belonging to the beginning of the Upper Palaeolithic (Aurignacian period). Generally, the earliest phase of Neolithic occupation should be roughly put in the second half of the 7th millennium BC (Early Neolithic). In particular, for Layer 9 of trench B, a charcoal sample has provided with a dating within the limits of the Early Neolithic period: 6200-6035 BC or 7261+20 BP (DEM-1211).

Trench C was opened alongside Trench A, with Layer 3 reaching to a depth of 0.50m and revealed a floor connected with Floor 4 found in Trench A (Layer 2). Pottery belonged to the Middle Helladic period, while a fair amount of obsidian fragments had been dumped here among soil debris of the early excavation works. These contained both pottery and obsidian fragments of the Late Neolithic Ib - Ia phases (Sampson, 2008).

The interconnection between the three trenches is not that simple as there is a lack (to our knowledge) of a satisfactory and efficient system of stratigraphic correspondence of respective dated layers and cultural phases. The exact correspondence has to be deduced from the edited publications (Sampson, 1998, 2000, 2008).

The burnt layers at a stratum are evidence of man's entrance into the cave towards the end of the Palaeolithic or the beginning of the Mesolithic Age, as proved by three absolute dating given by charcoal samples from this stratum (of Trench B) for layers and squares (rectangles).

Below the methods and sample preparation for mollusks are described in relation to stratigraphy and typological and radiocarbon dates, as well as the measured isotopic ratios. Then the OSL dating sample preparation, total dose and dose rate evaluation and the age result is discussed.

4. MOLLUSKS, SAMPLE PREPARATION AND DATA

Bivalve Shells

Samples of bivalve *Unio sp*. shell fragments (not properly studied) were measured by the isotope ratio mass spectrometer (Finnigan MAT with a Kiel II device). Measurements were examined at the Institute of Geology and Paleontology at Karl- Franzens University, Graz, Austria (Dr. Ana-Voica Bojar). For the sample amount, as little as 0.02–0.2mg equivalent carbonate in the form of powder was used. Values of $\delta^{18}O$ and $\delta^{13}C$ are given in per mil (‰) relative to PDB [δ per mil = [(Rsa/Rst) - 1] x 1000; R = the isotopic ratio; sa = sample; st = standard]. The internal CO_2 reference gas was calibrated against the NBS-18 standard.

CO_2 was extracted from the shells at 70 °C by reaction with H3PO4 (McCrea, 1950) using a Finnigan MAT Kiel II preparation line and an Finnigan MAT Delta Plus. Data were corrected for fractionation using the carbonate-phosphoric acid fractionation factors for calcite (Swart et al., 1991).

Twelve samples of lacustrine mollusc shells (*Unio s*p. tests) were obtained in sections A, B and C, ranging from depths of 0.90m down to 2.74m. Fragments of tests have been submitted to stable isotope analyses (^{18}O and ^{13}C). Dating of strata has been conformably provided according to archaeological chronological sequence stratigraphy, as well after absolute ^{14}C dating (yrs BP).

Sampling location intervals (depth, section, stratum and layer, square) and dating are presented in Table 1a,b. Numbering of material has followed the Archaeometry laboratory in Rhodes regulation standards (RHO_No_), whether samples have not been in form of powdered material to avoid any contamination that could affect measurements.

[1] *Unio* is a genus of medium-sized freshwater mussels, aquatic bivalve molluscs in the family Unionidae, the river mussels.

Table 1. a) Sarakenos cave mollusk shell sampling for δ18O, δ13C. Strata, layers, squares and section sampling intervals are shown. Dating (yrs BC) is according to archaeological findings and follow archaeological phases division known in the Aegean.

ARCH LAB_ RHO NO	Unio sp. Sample sorting	Section	Stratum layer	RSquare	Dating (BP)	Dating (BC)	Archaeological phases
RHO_21	1	C	6	2	-	2000-1800	Middle Helladic
RHO_22	2	C	9	5	3859±26	2400-2210	Early Helladic
RHO_23	3	A	5	4	-	2600-2400	Early Helladic
RHO_24	4	A	7	7	-	4200-4000	Late Neolithic IIa
RHO_25	5	C	11	8	-	4400-4300	Late Neolithic Ib
RHO_26	6	A	9	5	-	4600-4500	Late Neolithic Ib
RHO_27	7	C	16	4	-	4800-4700	Late Neolithic Ib
RHO_28	8	C	13	1	5931+25	4840-4730	Late Neolithic Ib
RHO_29	9	A	14	13	6125+42	5200-4960	Late Neolithic Ia
RHO_30	10	A	10	6	-	5200-5000	Late Neolithic Ia
RHO_31	11	B	15		6252+22	5300-5160	Late Neolithic Ia
RHO_32	12	A	13	8	-	5300-5200	Late Neolithic Ia
RHO_33	13	A	20	7	6794+21	5710-5650	Middle Neolithic

Table 1. b) Laboratory code number, along with archaeological and radiocarbon age, archaeological phases and isotopic ratios.

ARCH LAB_ RHO NO	Depth (m)	Dating (BP)	Dating (BC)	Archaeological phases	δ18O (PDB) *Unio sp.*	δ13C (PDB) *Unio sp.*	14C Dating (BP)	Arch Dating (BC)
RHO_21	0.90-1.12		2000-1800	Middle Helladic	-3.08	-2.16	-	2000-1800
RHO_22	1.05-1.35	3859±26	2400-2210	Early Helladic	-5.93	-2.97	3859+26	2400-2210
RHO_23	0.95-1.20		2600-2400	Early Helladic	-4.57	-1.98	-	2600-2400
RHO_24	1.40-1.60		4200-4000	Late Neolithic IIa	-5.33	-0.93	-	4200-4000
RHO_25	1.45-1.50		4400-4300	Late Neolithic Ib	-4.58	-3.60	-	4400-4300
RHO_26	1.68-1.80		4600-4500	Late Neolithic Ib	-6.07	-3.19	-	4600-4500
RHO_27	2.00-2.25		4800-4700	Late Neolithic Ib	-6.19	-1.40	-	4800-4700
RHO_28	1.65-1.80	5931±25	4840-4730	Late Neolithic Ib	-4.89	-0.85	5931+25	4840-4730
RHO_29	2.30-2.40	6125±42	5200-4960	Late Neolithic Ia	-6.73	-2.53	6125+42	5200-4960
RHO_30	1.80-1.95		5200-5000	Late Neolithic Ia	-5.86	0.98	-	5200-5000
RHO_31	1.87-2.04	6252±22	5300-5160	Late Neolithic Ia	-7.02	1.51	6252+22	5300-5160
RHO_33	2.45-2.74	6794+21	5710-5650	Middle Neolithic	-4.51	-1.68	6794+21	5710-5650

5. OSL DATING OF BURNT SOIL (HEARTH)

Samples and equipment

The brittle burnt earth was prepared in a powder form and sieved, without using water, to recover the 90-250μm grain sizes (the rest of the sample was used for dose rate measurements). These fractions were then cleaned in 10% H_2O_2 and 10% HCl to remove organics and carbonates, etched in concentrated HF for 40 min to remove any feldspar and finally washed again in 10% HCl.

Measurements were made in an automated Risoe TL/OSL reader equipped with blue LED (~50mW/cm² at 470-300nm) and IR laser (~500mW/cm² at 830nm) stimulation sources. Luminescence is detected though 9mm of U-340 filter, and the reader is fitted with an internal $^{90}Sr/^{90}Y$ beta source. Grains were mounted on stainless-steel discs using silicon spray as an adhesive. For the measurements 8 mm aliquots were used.

Total Dose evaluation

The single-aliquot regenerative-dose (SAR) protocol was used for the total equivalent dose for the separated quartz grains, in the procedure described in an earlier paper by Liritzis et al., 2015. The outline of SAR measurements is summarized in Table 2.

TABLE 2. Outline of the SAR Protocol to Sarakenos burnt soil.

1. Preheat of Natural (200°C)
2. Measurement of Natural Blue OSL Response (L_N)
3. Fixed Test Dose (10 Gy)
4. Heat Test Dose (160°C)
5. Measurement of Test Dose Blue OSL Response (T_N)

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6. Regenerative Dose
7. Preheat of Regenerative Dose (200°C)
8. Measurement of Regenerative Dose, Blue OSL Response (L_i)
9. Fixed Test Dose (10 Gy)
10. Heat Test Dose (160°C)
11. Measurement of Test Dose Blue OSL Response (T_i)

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12) Repeat steps 6) to 11) for a range of Regenerative Doses, 10, 20 and 40 Gy, including a Zero Dose and a Repeat point.

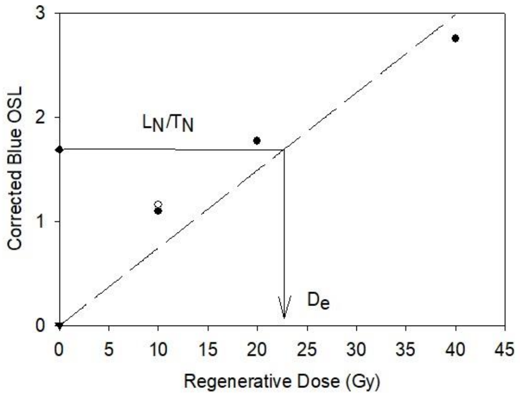


Figure 5: Graphical presentation of the SAR equivalent dose determination (sequence DB048, sample 6). The sensitivity corrected natural Blue OSL response (L_N/T_N – diamond), the sensitivity corrected regenerative dose points (L_i/T_i, i = 1, 2, 3 – solid circles), the zero point (L_i/T_i, i = 4 – triangle) and the repeated point (L_i/T_i, i = 5 – open circle) are plotted. The equivalent dose (D_e) is obtained by projection of the sensitivity corrected natural value onto the sensitivity corrected dose response curve.

In the protocol proposed here the Blue signal is measured for 50s at 125°C. The signal used is the Blue OSL signal detected in the initial 1 s of measurement. From this a background signal is subtracted based on the average Blue OSL signal observed in the last 5s of stimulation. The Test Dose measurement is used to correct sensitivity changes, by dividing the Blue OSL response to the subsequent test dose (L/T). The equivalent dose determination is shown graphically in Fig. 5.

Dosimetry

High resolution gamma spectrometry was used to provide the dosimetric information summarised in Table 3. The grain sizes that were left from the sieving were used to measure the gamma dose rate. For that measurement the soil is grinded and dried in an oven for 24h at 600°C. The material is then cast using polyester resin in a mould of 201cm^3. A cross section of sample mould is shown in Fig. 6. The measurement is based on the equilibrium of the ^{210}Pb with the ^{226}Ra of the uranium series. Storage for 23 days of the sample can be used to guaranty this equilibrium. Since the signal comes entirely from quartz, we assume that the alpha dose rate is negligible. With this equipment we are also capable of measuring the beta dose rate of the sample.

Table 3: Summary of dosimetry of burnt soil

Sample	Beta Dose rate, Gy/ka	Gamma Dose rate, Gy/ka	Cosmic Dose rate, Gy/ka	Beta Dose rate after water uptake correction, Gy/ka	Beta Dose rate after grain size correction, Gy/ka	Gamma Dose rate after water uptake correction, Gy/ka	Total Dose rate, Gy/ka
SARKN	1.25±0.09	0.67±0.03	0.13±0.025	0.87±0.06	0.81±0.07	0.48±0.02	1.52±0.08

To correct the Gamma Dose Rate considering the water content the equation $GD_C = 1/(1+((w.c.\%)/100*1.14))*GD$ was applied (where, GD gamma-ray dose rate and w.c the percentage of water uptake). For the corrected Beta Dose Rate (DB_c) the equation is $BD_C = 1/(1+((w.c.\%)/100*1.25))*BD$ (water content 25%, and the geometric factor of DG for the sample is considered 4π i.e. spherical). The correction factor for the specific grain size that is 0.93±0.05. An amount of 0.1 Gy/ka is added as an estimate of the internal dose-rate derived from the internal activity of clean quartz (Vagn Mejdahl measured this, years ago, for some representative samples, and came up with the number in the spreadsheet) (Liritzis, 2010). Cosmic ray dose rate is estimated 0.13 Gy/ka for the location deep in the cave (Prescott and Hutton, 1988).

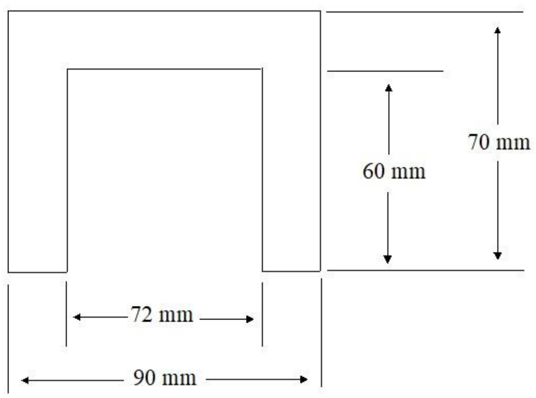


Figure 6: Cross section of sample mould

The equivalent dose (De) and the OSL age for sample SARKN provides the age of this burnt soil (Table 4).

Table 4: OSL age (from 19 single aliquots)

Sample	SARKN
D_e (Gy)	18.4±0.6 (n=19)
OSL age (ky), BP	**12.11±0.75**

Thus, the age of this burnt soil is 10,110±750 years BC and coincides with the onset of the interglacial period.

The OSL age result

Regarding the dating by ^{14}C on charcoal samples come from e.g. Layers B7 - R1, B9 -R2, and B9 - R3 in specific (DEM-1206, 9233±30 BP or 8530-8340 BC, DEM-1209, 9177±31 BP or 8450-8290 BC, and DEM-1210, 9230±30 BP or 8530-8340 BC).

The first and third dating come from the same hearth, with a characteristic absolute precision. The second one comes from another hearth and is slightly more recent. These ages are contemporaneous to the earliest Mesolithic phase in the Cyclops Cave (Sampson, 1998).

The sample of burned soil from the same hearth was dated with the method of OSL SAR technique above (initial preliminary report in Liritzis, 2008) provided a little higher age within the range of 9200-10700 years BC for a 65% confidence. This age consents with the upper limit of the radiocarbon ages. Consequently, despite the absence of Mesolithic finds, the presence of man inside the cave in this particular phase has been secured, and proved that the cave was used also in a late Mesolithic phase and later Neolithic periods.

6. X-RAY FLORESCENCE ANALYSIS OF CERAMICS AND SEDIMENTS

Provenance studies of the raw materials used during the prehistoric lithic industry are of key importance in research on ancient humans. Such studies provide information on the extension of the territory exploited by small groups of hunter–gatherers during the Palaeolithic. In the Neolithic and Bronze Age, provenance studies contribute to the knowledge of long-distance circulation and exchange of raw materials and goods, hence on the *chaines operatoires* of lithic and clay artefacts. Indeed, reconstructing mobility strategies is a major goal of researchers interested in prehistoric hunter–gatherers, and the use of geochemical source characterization of ceramics found at sites in a region offers a way to reconstruct the procurement range, or distance travelled by prehistoric groups to obtain resources.

Pottery, due to its remarkable storage properties, was a vital item used in the food activities of everyday life. Not only these uses, but aesthetic qualities, too, were frequently used by ancient humans. Ceramics are also preferred materials in provenance studies as their physico-chemical properties are most often different at a major, minor but mainly trace element level, because of their mode of formation from characteristic clay sources.

The standard statistical methods (hierarchical clustering, principal components analysis) and the developed model-based multivariate mixture of normals with an unknown number of components, are applied and compared (see a typical dendrogram in Fig.7).

The present new clustering approach was applied to a large data set initiated by the large Sarakenos analysed samples (details on statistical processing see Papageorgiou and Liritzis, 2007).

In fact, this work was initially thought to serve as a test rather than a provenance question, but the latter can't be excluded in the light of new finds. The data set under study consisted of 188 samples, derived from eight archaeological excavation sites of Mesolithic, Neolithic and Bronze Age: Hence, the Sarakenos cave in Boeotia, central Greece, was compared to ceramic fabric from Ftelia at Mykonos, Yali and Pergoussa near Nissiros (Dodecanese), Kalithies cave on the island of Rhodes, two settlements in Cyprus and Ulucak in Asia Minor near Smyrna.

The Sarakenos ceramics came along the excavated Trenches and cover the whole period from Neolithic to Bronze age layers.

Although distant sites (e.g., Boeotia and Cyprus) may exclude any possible contact, in spite of the overlapping period, nevertheless cultural contact between Neolithic and Bronze Age sites in Asia Minor and the Aegean has been documented. Evidence from settlements and specific areas involved include Youra, Euboea, Skyros, Boeotia, Nea Makri Attica, Tigani at Samos, Vathi in Kalymnos, Ulucak central Anatolia, St. George's cave Kalithies Rhodes and Agio Gala Chios (Furness, 1956; French, 1965; Hood, 1981; Kamil, 1982; Özdogan and Pendik, 1983; Sampson, 2006).

Moreover, Aegean population theories since the onset of the Holocene, assigning human movements from the Orient and the Balkans are also proposed (Ammerman and Cavalli-Sforza, 1984; Cherry, 1985, 1990; Van Andel and Runnels, 1988; Runnels, 1995; Broodbank, 2000).

Various practical matters concerning seafaring have affected the navigation, exploration and colonization of the Aegean. The travelling of long distances through navigation is not a surprise. Melian obsidian has been found in the cave of Cyclops in Youra, northwest Aegean, some 300 km from Melos, and at Maroulas Mesolithic site in Kythnos (Sampson et al., 2002), indicating a knowledge of navigation in the Aegean as early as the Holocene (Keegan and Diamond, 1987; Davies, 1992). In addition, the stone industry at Youra shows such contacts/similarities with other Anatolian caves, while the most recent field work (summer 2005) has revealed three Mesolithic sites on Icaria island, off the Anatolian coast, where stone artefacts have many similarities with those of Maroulas in Kythnos (Sampson and Koslowski, 1999; Sampson et al., 2010).

On the other hand, several similarities have been observed between western Asia Minor and the Aegean during the 9th to 7th millennia B.C., which are synchronized away from south-east Anatolia. Thus, at Dodecanese, the pottery shapes and decoration of 6th to 5th millennia BC Kalithies and Koumelo caves derive from various Anatolian and Aegean LN prototypes (Sampson, 1984; Melas, 1988), and from Melian obsidians found in east Attica caves implying Mesolithic seafaring activities (Laskaris et al., 2011).

Even if we exclude population movements between these distant islands by the sea, it would be realistic to argue a gradual spread of ideas through indirect contacts, taking into account the lower sea level by about 40–50 metres during the 9th to 7th millennia BC (Pirazzoli, 2000; Van Zeist and Bottema, 1982; Katsarou-Tzeveleki, 2001).

Cultural overlapping is a common image in the Aegean throughout its prehistory, underlying the significance of interaction and not necessarily revealing cultural domination from some adjacent cultures. Autochthonous and semi-autochthonous development seems to be the case of cultural interaction affected by local and inter-regional development.

With these points in mind, we attempted to cluster characteristic pottery finds from the aforementioned sites in the Aegean, western Asia Minor and Cyprus, even if the latter seems a remote possibility.

The ED-XRF field portable analyser Spectrace 9000 TN (Tracor Northern) was used with a mercuric iodide (HgI_2) detector, which has a spectral resolution of about 260 eV FWHM (Full Width Half Maximum) at 5.9 keV, and three excitation sources of radioisotopes within the probe unit: americium Am-241 (26.4 KeV K-line and 59.6 KeV L-lineV) measuring Ag, Cd, Sn, Ba, Sb; cadmium Cd-109 (22.1 K-line, 87.9 K- and L-line KeV) measuring Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Zr, Mo, Hg, Pb, Rb, Th, U; and iron Fe-55 (5.9 KeV K-line) measuring K, Ca, Ti, Cr. For the total number of samples (n=188) the analyser described above provided us with measurements of nine elements: Ba, Fe, Rb, K, Ti, Mn, Sr, Zr, Ca. The set of nine elements was the common subset of elements for which measurements were available for all the samples. The system was calibrated on several standard clays and bricks, and the application software 'Fine particle of soil application' was used.

Regarding Sarakenos cave ceramic and clays compared to the finds from other sites the following is asserted (Fig. 7) (see: Papageorgiou and Liritzis, (2007): 1) The Sarakenos cave group exhibits a greater spread around an apparent central nucleus, with an obvious 'outlier' SARA25, and others falling within neighboring clusters, e.g., SARA20 close to Kalithies Rhodes and SARA30 along the elongated distribution of the groups from Cyprus, and several RHO (Ulucak) groups (77, 86, 101, 83, 87, 75, 96) forming separate distinct subgroups within the SARA main cluster. Although some Ulucak sherds (RHO39 EBA, RHO107 LN, RHO80 LC, RHO81 LN, RHO92 LN) of Late Neolithic (LN), Early Bronze Age (EB) and Late Chalcolithic (LC) periods, overlap with some SARA groups (4 EBA, 29 EBA, 3 MN, 17 MN) from Early Bronze and Middle Neolithic, SARA42 of LN I a–b period belongs to the same subgroup as RHO: 72 LC, 95 LN and 69 LC. This interesting pattern implies possible interactions (exchange of ceramics and/or sharing the same clay source), enhanced by the fact they are of the same period, i.e., Late Chalcolithic/Early Bronze Age

(4000–2500 BC), Late Neolithic and Middle Neolithic. This finding needs further verification.

The extremely interesting Ulucak–Yali–Pergoussa and Sarakenos–Ulucak interactions should be verified further.

The detailed chemical analysis of the ceramics and sediments from Sarakenos Cave excavation is given in Sampson (2008).

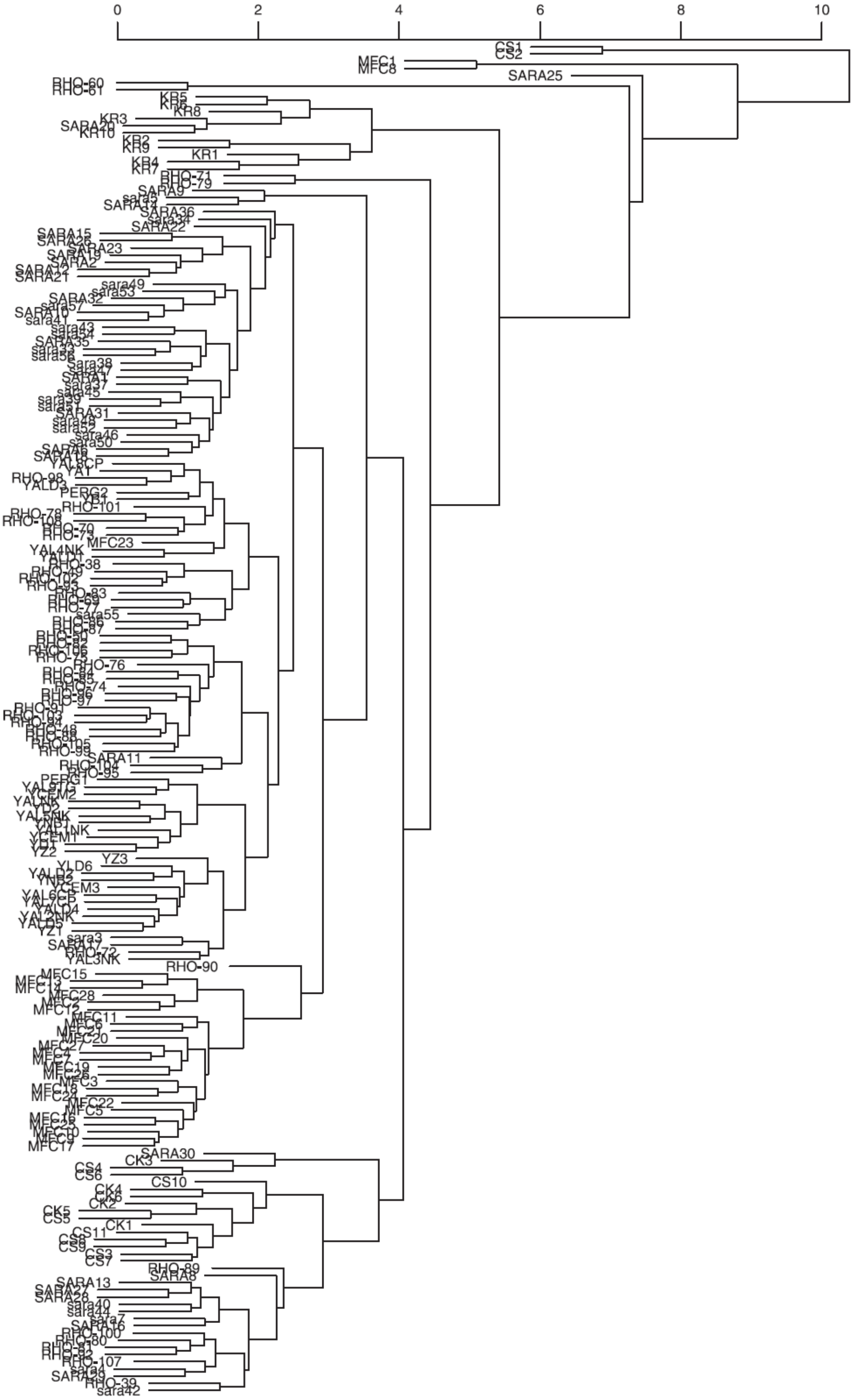


Figure 7. A dendrogram based on cluster analysis (hierarchical, average linkage) of the data set in total. The 188 samples were from Sarakenos cave and other sites (Aegean islands and Asia Minor). The main aim was to identify clear groups that imply provenance and in general trade interaction (Papageorgiou and Liritzis, 2007).

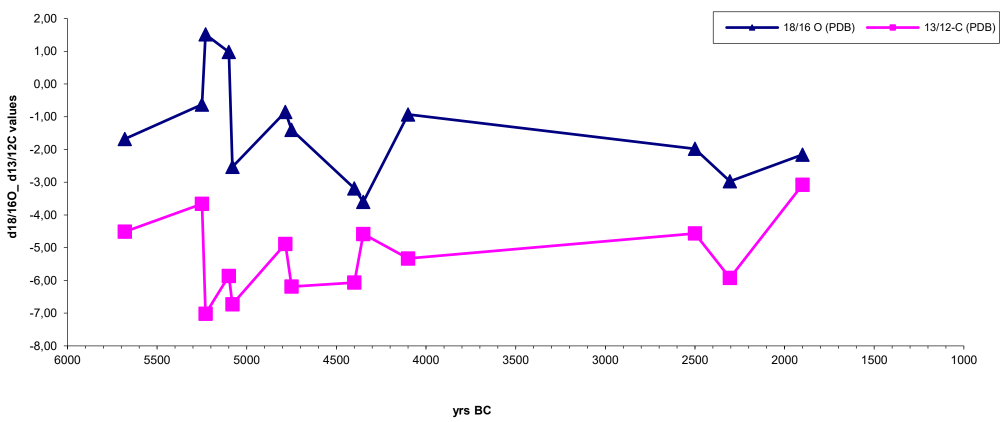
7. ISOTOPIC VARIATIONS

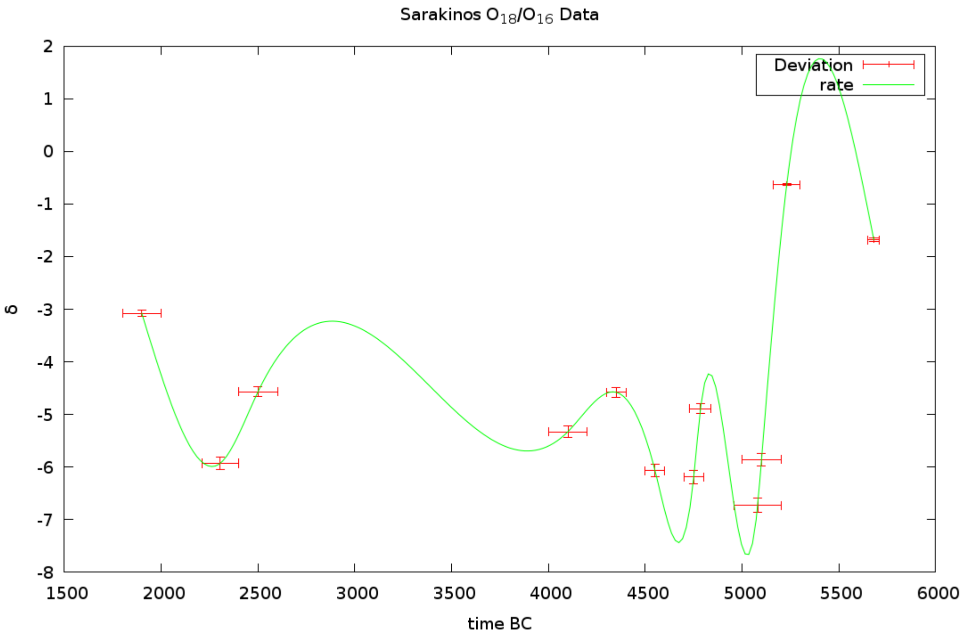
Fig. 8 gives the isotopic data of the freshwater molluscs of oxygen and carbon stable isotopes for the period ca.1500 to ca.5700 BC. Stable isotope ratios serve as climatic proxy, though any interpretation must include several data from environmental parameters such as burial conditions, stratigraphic documentation, accurate dating, dense data points.

In our preliminary work, with few data points we address the issues and comment with due caution.

The maximum deviated values for the available data points are 6‰ for carbon and 7‰ for oxygen. It is worth comparing these data with the partially overlapping and earlier period (ca.5000 to ca.8500 BC[2]) variation of marine and terrestrial molluscs of another cave at Alonissos, the Youra cave (Drivaliari et al., 2011), and elaborate on the interpretation of the results.

Sarakenos Cave: *Unio sp.* stable isotope analyses: d18/16O_d13/12C variations





[2] The calibration of the conventional radiocarbon dates was performed according to the Radiocarbon Calibration Program Rev. 4.3 (Stuiver and Reimer, 1993).

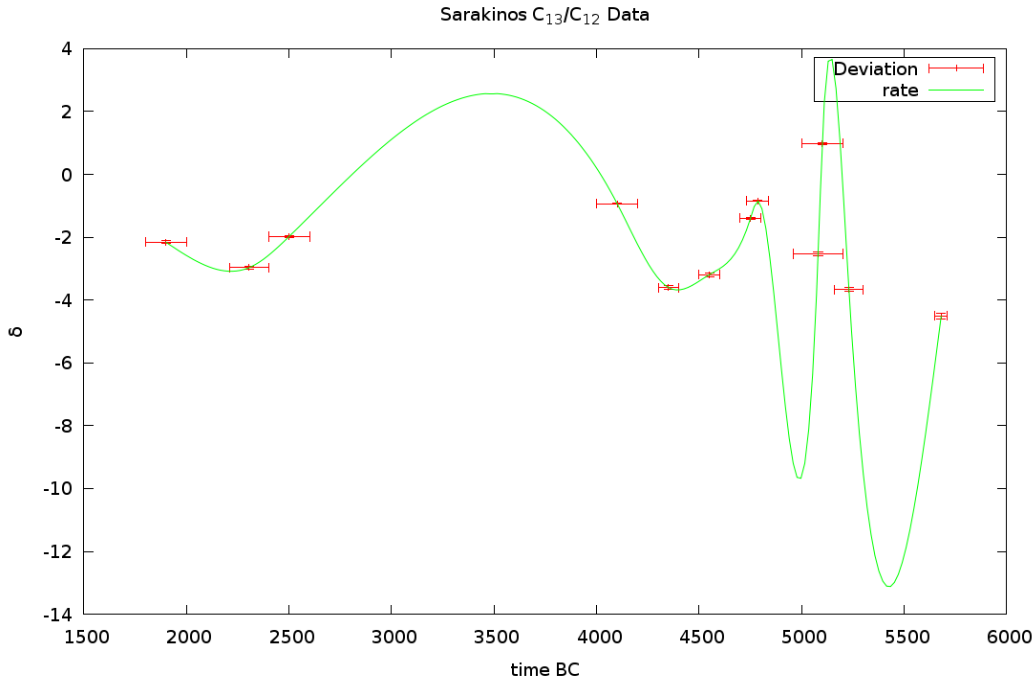


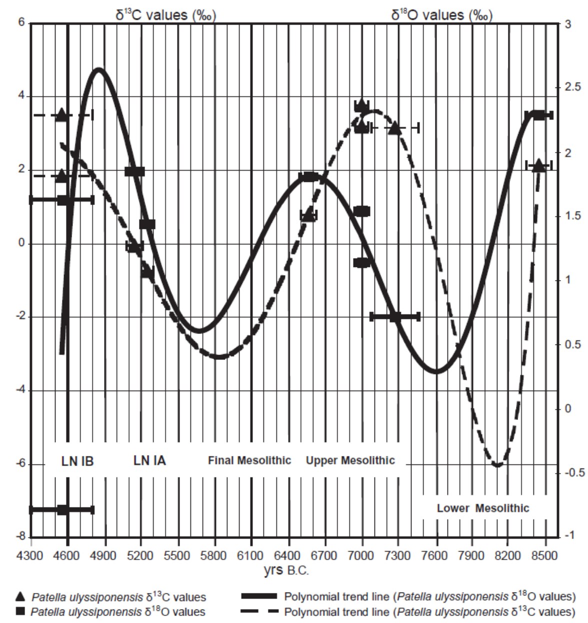
Figure 8. Isotopic values of fresh water molluscs Unio sp. of $\delta^{18}O/O^{16}O$ and $\delta^{13}C/^{12}C$ against time (years BC) (age ranges from ca.1000 to ca.6000 years BC). The lower plots present a polynomial best fitting to the data, as a best trend. More data especially ca.3500 and 5500 BC are needed to reinforce the curve.

Table 5. Variations of isotopic ratios per 5200-4500 BC. Numbers in per mil. (m: from the curve trend, model)

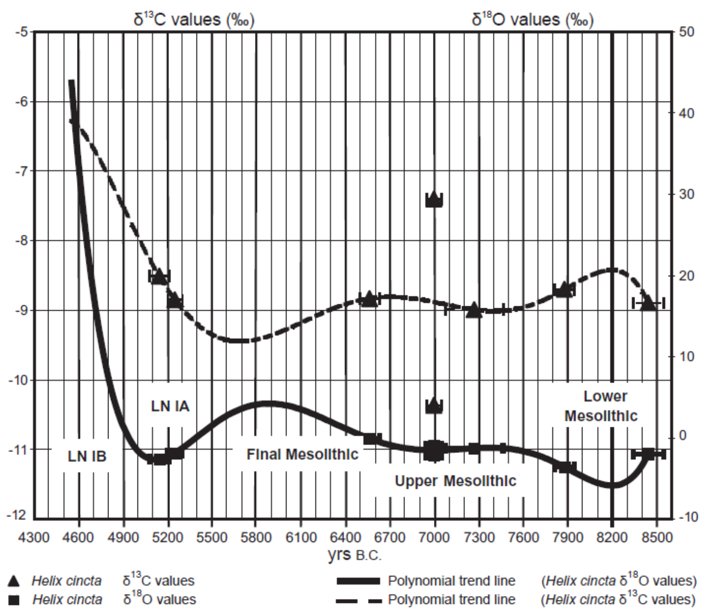
Site/5200-4500 BC	Carbon	Oxygen	Oxygen, av.	Carbon, av.
Sarakenos, freshwater	-3 to +1	-6 to 0 (m: -5 to +2)	-1.7 (5200-4500)	-5.5 (5200-4500)
Youra, marine	-3 to +1	+0.5 to +2 (m: 0.5 to +4)	+1 (4300-8500)	+2 (4300-8500)
Youra, terrestrial	-8.5/9	-11 (m: -6.5 to -11)	-11 (4300-8500)	-8.7 (4300-8500)

Table 5 gives the variation of the isotopes as averages over the whole respective interval, and in particular for the (though scarce) available data in the overlapping time segment 5200-4500 BC.

Inspection of Table 5 despite the scarce of data and taken into account error bars, the following general pattern emerges: 1) it appears that the two caves share similar climatic regime for the overlapping period of 5200-4500 BC, comparing Sarakenos freshwater and Youra marine molluscs especially for carbon ratios, but oxygen ratios trend, too (Fig. 9), while Youra terrestrial species obviously differ, 2) Isotopic ratios after ca the 6th millennium BC (~8000 BP) are negative, while earlier on at least for the data period covered in the present work (5500-8000 BC) the oxygen ratios most sensitive to temperature variations are more positive or closer to zero and terrestrial carbon ratios much negative, 3) the Oxygen and Carbon data for Sarakenos are commensurable but ca. 4300 BC the anticorrelation is puzzling, if it is due to wrong layer attribution of the sample.



(A) Marine shells



(B) Terrestrial

Figure 9. Isotopic values of fresh water molluscs Unio sp. of Cyclop's cave Youra, Alonissos, of $\delta^{18}O/^{16}O$ and $\delta^{13}C/^{12}C$ against time (A) marine and (B) terrestrial origin (from Drivaliari et al., 2011)

8. DISCUSSION ON ISOTOPIC DATA

Any Controls on the oxygen isotope ratios within molluscan carbonate, varies for each type of mollusc, with the environmental waters thought to be the most dominant influence. In fact, freshwater molluscs, where major controls are whether the lakes can be either closed (the signal will be evaporation) or open (the signal will be some aspect of precipitation). In both the Kopais lake and the Sarakenos cave applies the open system. Thus, the pronounced control in isotopic variation depends on amount, source, and temperature of precipitation (humidity) of the environmental waters.

In lakes the signal is often due to evaporation (terminal lakes) or precipitation in open lakes where water flows freely through it (see: Jones et al., 2002; Leng and Lewis, 2016; Wang et al., 2019).

Changes between -13‰ to -5‰ in ^{18}O imply mixed carbon pool of both dissolved inorganic carbon (high ^{13}C from -3 to +3‰) directly from ambient water and dietary organic carbon (primarily particulate algae and plant debris, values between -16‰ to -35‰). While for terrestrial and lung breathing freshwater molluscs, the major controls are evaporation, precipitation (humidity) and temperature, for marine molluscs, the major controls are salinity, temperature and over longer time scale the Global Ice Volume (see, Drivaliari et al., 2011); and for estuarine molluscs, the major controls are salinity and temperature (Leng and Lewis, 2014).

The use of oxygen isotope ratios ($^{18}O/^{16}O$, from which we derive $\delta^{18}O$ from molluscan carbonate is an established method to derive environmental information from daily (Goodwin et al., 2001) to centennial scale (Schöne et al., 2004; Scourse et al., 2006) time periods. In our present data we use whole shells thus integrating the whole growth period.

An example of seasonal $\delta^{18}O$ and $\delta^{13}C$ data from the shell of a fossil freshwater bivalve (Unio sp.) collected from Çatalhöyük, Central Anatolia, Turkey is given by Bar-Yosef Mayer et al. (2012). This bivalve likely lived between 4 and 5 years in ca. 8500 cal years BP, providing insight into annual climate cycles during the early Holocene (see annotations) during the occupation phase of Çatalhöyük.

Rising ^{18}O values towards more negative (lowest) ^{18}O imply warmer years due to evaporation. The lowest values usually occurring in the winter due to shell carbonate precipitation of isotopically low freshwater close to the mean –weighted annual precipitation i.e $\delta^{18}O$ =-12 / ‰. (Leng et al., 1999). Co-variation of $\delta^{13}C$ and $\delta^{18}O$ suggests that seasonal climate change might influence carbon source to some degree.

The $\delta^{18}O$ composition of the final shell growth has been used since the early work of Shackleton (1973) who showed that the last deposited carbonate (at the ventral margin in bivalves, and the aperture in gastropods) reflects the environmental conditions at the time of the animal's death, which in archaeological deposits is often assumed to be the season of collection, if a change in the environmental conditions occurs between seasons. This might be temperature in coastal settings, rainfall composition in terrestrial snails or summer evaporation in freshwater lakes.

In fact, seasonal $\delta^{18}O$ data from multiple fossil Unio shells from the world famous Çatalhöyük archaeological site in south central Turkey has shown that there was a reduction in seasonality at around 7,000 to 6,000 years BC. This trend coincides with other records of climate change at the same time, where there was a shift towards drier and cooler conditions and a reduction in winter precipitation and summer evaporation (Bar Yosef Mayer et al., 2012)

When studying past environments, we often use qualitative descriptions of the climate (e.g. 'it got warmer'; 'it got drier') and due to lacking present oxygen data to create a functional modelling only approximate climatic evaluation is possible.

Carbon isotope ratios ($^{13}C/^{12}C$, from which we derive $\delta^{13}C$) are collected alongside $\delta^{18}O$ from molluscan carbonate. In aquatic (marine and freshwater) environment,

CO2 and the dissolved inorganic carbon (DIC) ion are often the main source of carbon for Mollusca. Shell $\delta^{13}C$ is typically a few ‰ lower than ambient $\delta^{13}C$ DIC, which can reflect processes such as changes in salinity (in coastal and estuarine environments) (McConnaughey and Gillikin, 2008).

In terrestrial molluscs, $\delta^{13}C$ will be influenced by carbon from several sources including ingested organic matter and carbonates as well as atmospheric CO_2. Previous studies have mainly found that the primary variable affecting $\delta^{13}C$ in terrestrial snail shells is diet.

Therefore, in herbivorous species, $\delta^{13}C$ is often thought to be a proxy for changes in palaeovegetation mainly in terms of the distribution of C3 and C4 plants and changes due to water stress (Yapp, 1979; Francey, 1983; Goodfriend and Ellis, 2002; Stott, 2002; Metref et al., 2003; Balakrishnan et al., 2005a, 2005b; Baldini et al., 2007). Other studies have observed consistent inter-specific variability in $\delta^{13}C$ likely reflecting species-specific feeding behaviour (e.g. Goodfriend and Ellis, 2002; Colonese et al., 2010). In some calciophilous species, the contribution of soil carbonates to the diet seems to be important (Yanes et al., 2009).

Paleoecological data show a succession of dry events at 3800–3700, 3450 and 3000–2900 cal. yrs BC. These events correspond to incursion of cold air masses to the eastern Mediterranean, confirming the climatic instability of the middle Holocene climate transition. Two periods with farming and pastural activities (4300–3600 and 3100–2700 cal. BC) are evident. The intervening period is marked by environmental changes, but the continuous occurrence of anthropogenic taxa suggests the persistence of human activities despite the absence of archaeological evidence. The environmental factors alone were not sufficient to trigger the observed societal changes (Lespez et al., 2016).

The presence of the freshwater snails forms a strong indication that during the Chalcolithic period the climate was more humid than today (from the late Neolithic to the Early Bronze Age (6th – 3rd millennia

BC). The Chalcolithic snail fauna but forest too differs considerably from that of today due to ancient and recent anthropogenic changes in the land use with a similar phenomenon in Near East (Mienis et al., 2017).

9. CONCLUSION

A combined archaeometric investigation of shells, sediments, ceramics and burnt sediment from Sarakenos Mesolithic period cave provided interesting results. The characterization and provenance study of sediments along the trench and ceramics from Neolithic period to Bronze age compared with respective periods from other archaeological sites in the Aegean and Anatolia have indicated possible trade pattern. This is strengthening by obsidian trade and dating evidence of Mesolithic times navigation in the Aegean.

The OSL dating of burnt soil provided the commensurable age to archaeological record and C-14 ages of charcoal of ca 10,000 BC and implies man's entrance into the cave using hearth. The isotopic data of the freshwater molluscs of oxygen and carbon stable isotopes for the period ca.1500 to ca.5700 BC serve as climatic proxy, though any interpretation must include several data from environmental parameters such as burial conditions, stratigraphic documentation, accurate dating, dense data points.

The seasonal $\delta^{18}O$ data from multiple fossil Unio shells shows probably that there was a reduction in seasonality at around 7000 to 6000 years BC. This trend coincides with other records of climate change at the same time, where there was a shift towards drier and cooler conditions and a reduction in winter precipitation and summer evaporation. There are also indications of drier climate (and lake level drop) between 4000 BC and 2500 BC.

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