Analytical identification of a single source pumice from Greek shores and ancient sites in the Levant*

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ABSTRACT: Alluvial pumice beds from Greek shores were investigated by INAA and compared to the pumice database compiled in the course of the SCIEM2000 research programme. This database contains the compositional data on 25 quaternary volcanic eruptions in the Mediterranean region that produced relevant amounts of pumice. The so-called chemical fingerprint, consisting of the concentrations of the elements As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Nd, Rb, Sb, Sc, Sm, Ta, Tb, Th, U, Yb, Zn, and Zr is used to relate pumice to its source volcano. Thereby, a perfect compositional agreement of the Greek alluvial pumice with a number of pumice finds from archaeological excavations in the Levant was observed. Though the source volcano of this material is still unknown, it is demonstrated that the composition indicates a source in the Southern Hellenic island arc, probably in the neighbourhood of Milos.
Key-words: *Geochemistry, INAA, trace elements, pumice, provenancing*.

ΠΕΡΙΛΗΨΗ: Δείγματα, από στρώματα κίσηρης αλλουβιακής προέλευσης, από τις ακτές της δυτικής Πελοποννήσου, της δυτικής Κρήτης καθώς και από τα παράλια της νοτιοανατολικής Μεσογείου (Levant), αναλύθηκαν χημικά, με τη μέθοδο της νετρονικής ενεργοποίησης (INAA). Ακολούθως, έγινε σύγκριση της σύστασης τους με τη βάση δεδομένων για κίσηρη που είχε δημιουργηθεί στο πλαίσια του ερευνητικού προγράμματος SCIEM2000. Η βάση αυτή, περιέχει δεδομένα για τις συστάσεις της κίσηρης που έχει δημιουργηθεί από 25 διαφορετικές τεταρτογενείς ηφαιστειακές εκρήξεις στην περιοχή της Μεσογείου.

Το λεγόμενο χημικό δακτυλικό αποτύπωμα, το οποίο συνίσταται στις συγκεντρώσεις των στοιχείων As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Nd, Rb, Sb, Sc, Sm, Ta, Tb, Th, U, Yb, Zn, και Zr, χρησιμοποιήθηκε για να προσδιοριστεί η προέλευση των δειγμάτων της κίσηρης.

Η γεωχημική μελέτη απέδειξε μια τέλεια συμφωνία (έως ταύτιση) στη σύσταση τόσο των δειγμάτων της κίσηρης, από τα παράλια της δυτικής Πελοποννήσου και της δυτικής Κρήτης όσο και μιας σειράς δειγμάτων από τις αρχαιολογικές ανασκαφές στα νοτιοανατολικά παράλια της Μεσογείου (Levant).

Αν και η πηγή προέλευσης αυτού του ηφαιστειακού υλικού δεν έχει ακόμη εντοπιστεί με ακρίβεια, η χημική σύστασή του, συνηγορεί για μια ενιαία ηφαιστειακή πηγή στο νότια τμήμα του ηφαιστειακού τόξου του Αιγαίου, πιθανώς στην περιοχή της Μήλου.
Λέξεις-κλειδιά: *Γεωχημεία, ανάλυση με νετρονική ενεργοποίηση, ιχνοστοιχεία, κίσηρη, ηφαιστειακό τόξο νοτίου Αιγαίου*.

INTRODUCTION

The long-term investigations of volcanic material found in the context of archaeological excavations in the eastern Mediterranean region allowed to identify the sources of hundreds of pumice finds by comparison of their compositions, the so called "chemical fingerprinting". These investigations and the compilation of the analytical data base on Mediterranean pumice sources have been carried out in worldwide cooperations in the framework of the special research programme SCIEM 2000, the "Synchronization of Civilizations in the Eastern Mediterranean region in the 2nd Millennium BC. The results were obtained by neutron activation analysis (NAA) and compiled in several publications (Peltz *et al.*, 1999; Steinhauser *et al.*, 2006b; Steinhauser *et al.*, 2007). NAA was chosen because it allows to simultaneously determine a large number of geochemically significant elements in very small samples (Steinhauser *et al.*, 2006a) with adequate accuracy and precision. The most recent analytical work and some exemplary archaeological details are given in Steinhauser *et al.* (2010). The identification of volcanic material enables to backtrack transport routes for natural as well as for trading connections. An additional chronological aspect stems from the fact that for a known date of the respective eruption, pumice can act as a time mark, giving a *datum post quem* for the deposition of the pumice bearing strata.

However, in the course of this SCIEM2000-related research, a few archaeological samples remained unidentified, but attracted attention due to their highly similar composition. These pumice finds were unearthened at the famous sites of Tell-el-Dabᶜa (the ancient Hyksos capital Avaris) in the Nile Delta and at Ashkelon (Palestine). The respective strata were dated to the Middle Bronze Age, one to the Iron

* Προσδιορισμός με αναλυτικές μεθόδους της κοινής προέλευσης στρωμάτων κίσηρης που έχουν εντοπιστεί στις ακτές της δυτικής Πελοποννήσου και δυτικής Κρήτης, καθώς και σε αρχαιολογικούς χώρους, στα νοτιοανατολικά παράλια της Μεσογείου (Levant)

Age, and one remained undatable. With this background, the discovery of pumice bearing coastal sediment beds in the Gulf of Kyparissia, western Peloponnesus and the first analytical results (BATHRELLOS *et al.,* 2009) led to a cooperation between the Faculty of Geology & Geoenvironment, (National and Kapodistrian University of Athens) and the Atominstitut (Vienna University of Technology). Comparative analyses were carried out in order to identify the source volcano via the SCIEM2000 database. Additionally, the accuracy and reproducibility of the different analytical methods were checked. Pumice samples from a newly discovered pumice-bearing bed, located in northwest Crete Island (Kissamos bay) were added to this investigation. With the first analytical run it was clear that the alluvial pumice lumps from Kyparissia and Crete belong to the same compositional group as the unidentified archaeological samples mentioned above. The aim of this work is to narrow down the range of the possible source volcanoes. The geographical situation of the find spots is presented in Fig. 1, a photographic overview for the pumice finds from Kissamos bay is given in Fig. 2.

SAMPLING

Pumice is a highly vesicular volcanic eruption product that consists mainly of foamy silicate glass and floats on water due to its low bulk density. This property leads to wide-spread pumice distribution by marine currents and wind when erupted from sources close to the sea or even submarine volcanoes (e.g. SUTHERLAND, 1965). Accordingly, floating pumice accumulates along the shorelines and in some cases, workable deposits like that near Ayia Irini (Cyprus) were formed (MOORE, 1960). As pumice is a useful abrasive, pumice deposits have been exploited and products like lumps and powder traded since prehistoric times. This is known from descriptions of the various applications by the roman author C. Plinius Secundus (PLINIUS SECUNDUS, † 79 A.D.) as well as by findings from archaeological excavations (see e.g. FAURE, 1971; WARREN & PUCHELT, 1990; PELTZ & BICHLER, 2001; HUBER & BICHLER, 2003; STEINHAUSER *et al.*, 2006b).

The Kyparissia pumice bed is described in BATHRELLOS *et al.* (2009), as a pumice-bearing horizon of about 10 cm, lying in a depth of 30 cm below siliceous sand. It consists of mainly flattened pumice pebbles with sizes from 0.2 cm to 6 cm in diameter and, rarely, bivalve shells (pectinidae).

The Kissamos bay pumice deposit is hosted in a reddish unconsolidated bed that is composed of clayey materials, angular fragments of the Mesozoic substrate and older beach rocks, as well as the rounded pumice pieces [varying in size from <1 cm up to 10 cm], and overlies paleosol and beachrock formation. It lies some 50 m from the present day littoral line and in an altitude of ~10 m above sea-level. The outcrop is lensoid and up to 20 cm thick, thinning southwards and northwards.

In Milos Island the early volcanic activity (Middle–Late Pliocene) has produced a thick (>120 m) succession of felsic, pumice-rich, submarine units. Moreover, the recent volcanism (Late Pleistocene to Present) that has been subaerial, has formed two rhyolitic pumice cones among the other volcanic

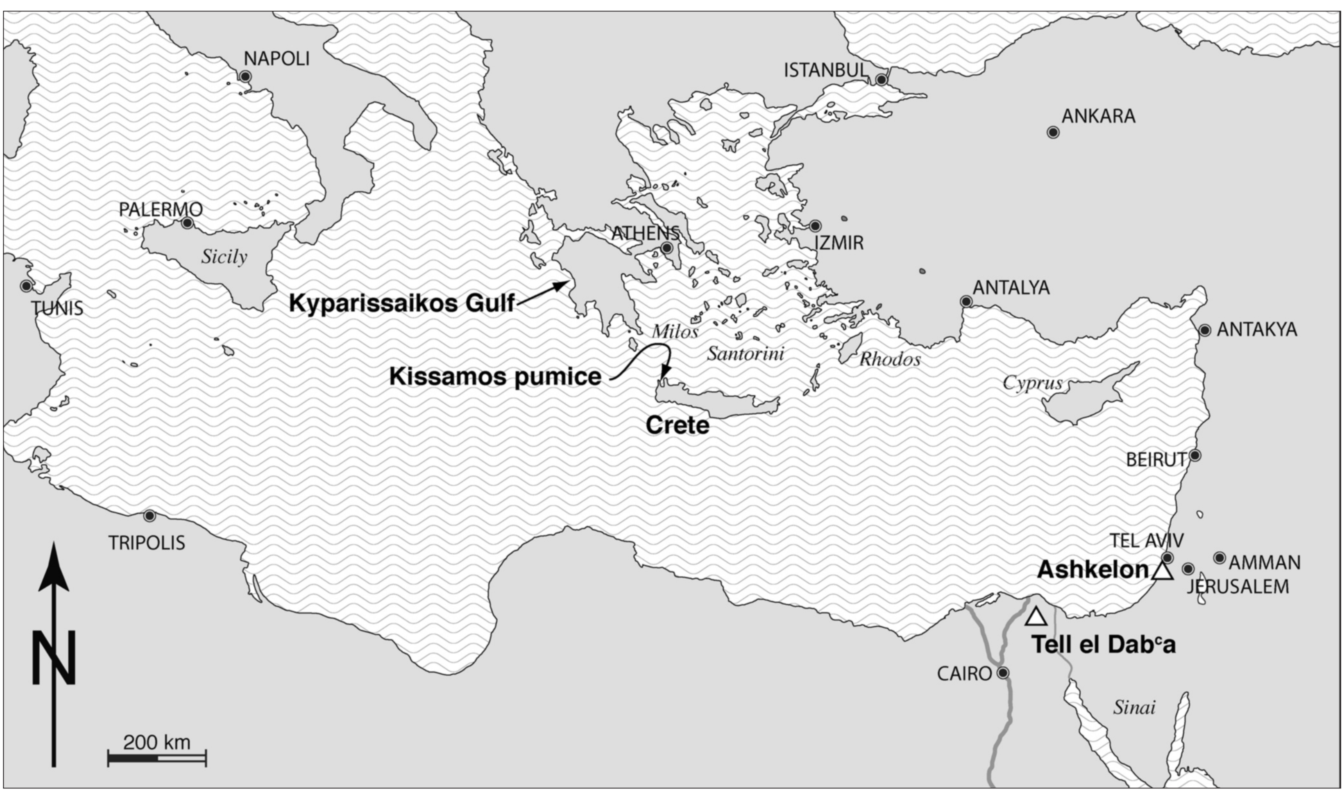

Fig. 1. Geographical situation.

Fig. 2. Pumice bearing sediment at the Kissamos bay (Crete). The reddish bed is the one that hosts rounded pieces of pumice, externally stained by iron oxides.

rocks (Fytikas *et al.,* 1986). Sampling has been performed at several places coverimg the prominent pumice outcrops near Plakes, Mavrovouno (1 km east of Ag. Sofia), Sarakiniko beach, Papafrangas and Trachilas. A detailed description can be found in Peltz *et al.* (1999) and Peltz (1999). For analysis only lumpy pumice or pumice lapilli were used to limit the investigation to the fresh magmatic components of the respective eruptions.

ANALYTICAL TECHNIQUES

From the hosting formations at Kissamos Bay and the Kyparissaikos Gulf, pumice pebbles ranging in size between 0.2 cm and 6 cm were sampled. Consequently, the pumice pebbles were washed-out by deionized water. Mineralogical, textural and microprobe analysis of the pumice pieces were performed in thin sections, using an optical microscope and a scanning electron microscope at the laboratories of the NKUA, Geology and Geoenvironment Faculty (JEOL JSM-5600 equipped with Oxford Link ISIS 300 Energy Dispersive microprobe analytical system, beam current: 0.5 nA and 2 μm diameter).

In addition, Instrumental Neutron Activation Analysis (INAA) was applied to determine the major and trace element abundances. In particular, the elements As, Ba, Ce, Co, Cr, Cs, Eu, Fe, Hf, K, La, Lu, Na, Nd, Rb, Sb, Sc, Sm, Ta, Tb, Th, U, Yb, Zn, and Zr were determined. The applicability of this technique for the distinction of chemically rather similar eruption products of various pumice sources, such as Santorini, Kos, Giali, Nisyros, and Milos from the southern Hellenic volcanic island arc, Lipari (Aeolian islands) and Cappadokia has been demonstrated in earlier studies (Peltz *et al.,* 1999; Bichler *et al.,* 2004; Steinhauser *et al.*, 2006b; Steinhauser *et al.*, 2007). The first application to archaeologically stratified pumice from excavations at Tell-el-Dab^{c}a (Egypt) showed that even long storage under wet conditions as in the Nile-Delta sediments does not affect the proper classification by chemical fingerprinting (Peltz & Bichler, 2001), although chemical adsorption reactions do take place under certain conditions (Steinhauser & Bichler, 2008; Sterba *et al.*, 2008).

All samples were prepared in our routine analytical procedure for pumiceous material including a thorough cleaning procedure in distilled water in an ultrasonic bath, microscopical investigations, and homogenization of a representative amount using agate mortar and pestle. Quantities of about 100 mg of each sample were weighed into Suprasil™ quartz glass vials, sealed, and irradiated together with internationally certified standard reference materials in the neutron flux of the TRIGA Mark II reactor at the Atominstitut in Vienna. The multielement standards used for the quantitative analysis were the CANMET reference soil SO1, NIST SRM 1633b (Coal Fly Ash), BCR No. 142 (Light Sandy Soil), NIST SRM 2702 (Inorganics in Marine Sediments) and the MC rhyolithe GBW 07113. After decay times of 5 days and 4 weeks, respectively, the activation products were measured by γ-spectrometry and the concentrations of the respective elements were calculated.

RESULTS AND DISCUSSION

The analytical results are presented in Table 1. Fig. 3 shows the perfect agreement with data from the literature by displaying "chemical fingerprints" (Bathrellos *et al.,* 2009). These element distribution patterns are obtained by normalisation of the measured concentrations to those in the upper crust, as given by Taylor & McLennan (1985). Bathrellos *et al*. (2009) made the assumption that the source of the material could be from the western part of the southern Aegean volcanic arc, at or near the island of Milos. This assumption is corroborated by the fact that the composition falls clearly in the range observed by Peltz *et al*. (1999) for selected pumice from Milos. Fig. 4 shows this compositional range in comparison to the alluvial samples found in Kyparissia and Crete. However, none of the 14 single pumice patterns that form the overall range for Milos exactly matches the alluvial samples. To demonstrate this misfit and the effectiveness of the identification method by chemical fingerprinting, Fig. 5 shows a comparison of the alluvial samples to a set of 8 pumice samples obtained from the Sarakiniko beach outcrop. The pumice-bearing layers have been sampled all over the full thickness of this deposit. For analysis, only the pure pumice resembling the juvenile magmatic component has been used. The extraordinary homogenity of this material as well as the precision of the data can be recognised at one glance. For sampling and preparation details see Peltz *et al*. (1999).

Fig. 6 contains the fingerprints of 13 pieces of pumice found in the archaeological excavations at Tell el Dab^{c}a and Ashkelon and demonstrates their most remarkable similarity with the alluvial samples from Kyparissia and Crete. The outliers in Sb and As are explained by local anthropogenic contaminations and have been observed also at other occasions

TABLE 1

Element concentration (mg/kg) of pumice analyzed in this study and literature values used for comparison. The analytical error is <10 % for the elements Cr, Nd, Sm, Lu, and U and <5 % for all other elements.

sample	location	Na	K	Sc	Cr	Fe	Co	Zn	As	Rb	Zr	Sb	Cs	Ba	La	Ce	Nd	Sm	Eu	Tb	Yb	Lu	Hf	Ta	Th	U
MLO 2	Plakes	28500	32900	2,74	2,5	8720	0,88	37	4,6	130	123	0,89	3,78	555	27,2	47	16	3,2	0,524	0,5	2,2	0,37	3,62	0,69	14,98	3,51
MLO 3	Trachilas	26700	39500	4,14	n.d.	5920	0,43	40	2,8	152	103	0,29	4,77	473	26,5	52	20	4,4	0,300	0,7	4,1	0,68	3,00	1,55	19,87	5,21
MLO 4	Mavrovouni	30400	35700	2,66	3,0	8810	1,10	40	4,0	111	114	0,30	3,94	492	24,1	43	16	3,1	0,438	0,4	2,1	0,35	3,34	0,72	12,43	3,19
MLO 5	Plakes	25300	32300	1,59	0,7	7740	0,58	32	2,3	119	119	0,21	3,70	516	26,0	48	16	3,0	0,469	0,4	2,1	0,37	3,21	0,81	14,78	3,85
MLO 6	Sarakiniko	28200	37300	1,68	1,9	6640	0,56	31	1,7	130	119	0,16	2,39	661	33,1	54	15	3,0	0,406	0,4	1,8	0,31	2,92	1,12	19,25	4,58
MLO 7	Sarakiniko	29300	37900	1,55	2,5	6830	0,51	31	2,1	124	118	0,20	2,41	711	33,5	56	18	3,1	0,417	0,4	1,9	0,33	2,98	1,15	19,71	4,80
MLO 8	Sarakiniko	24100	39700	1,66	0,8	6070	0,37	28	1,6	128	109	0,15	2,36	668	33,0	54	16	3,0	0,369	0,4	1,8	0,32	2,92	1,14	19,59	4,69
MLO 9	Sarakiniko	26800	38700	1,55	1,4	7030	0,42	30	1,2	124	121	0,21	2,29	684	34,3	56	16	3,2	0,449	0,4	1,8	0,33	3,07	1,13	19,50	4,62
MLO 10	Sarakiniko	28200	35600	1,52	1,4	6760	0,39	30	1,1	122	121	0,17	2,28	676	33,6	55	16	3,0	0,427	0,4	2,0	0,32	3,09	1,11	19,04	4,56
MLO 11	Sarakiniko	34400	38400	1,49	1,6	6690	0,38	30	1,0	121	118	0,17	2,31	675	33,3	55	16	3,0	0,429	0,4	1,8	0,32	3,02	1,09	19,22	4,61
MLO 12	Sarakiniko	24100	39300	1,53	1,3	6870	0,55	31	1,5	126	106	0,18	2,35	676	34,0	55	15	3,0	0,421	0,4	1,8	0,32	2,99	1,12	19,45	4,69
MLO 13	Sarakiniko	21400	36300	1,77	1,3	6970	0,66	30	1,4	126	117	0,24	2,34	670	33,4	55	15	3,0	0,410	0,4	1,9	0,32	3,08	1,12	19,65	4,84
MLO 14	Papafrangas	11700	18400	1,36	n.d.	4550	0,45	20	2,6	83	91	0,94	3,21	548	15,0	26	11	2,2	0,290	0,3	1,2	0,21	1,88	0,34	7,64	6,10
MLO 15	Papafrangas	11100	19800	1,45	0,7	4830	0,45	21	2,3	90	116	0,88	3,33	622	16,4	30	13	2,6	0,311	0,3	1,3	0,23	2,12	0,38	8,62	7,56
alluvial pumice																										
2009/01A	Crete	32600	33800	1,88	6,3	12700	1,4	54	2,5	116	128	0,21	2,79	460	31,2	57	19	3,8	0,522	0,5	2,9	0,42	3,90	1,49	14,29	3,69
2009/01B	Crete	34400	35100	1,17	1,8	11400	0,4	52	2,0	123	128	0,16	2,81	487	32,0	58	19	3,7	0,481	0,6	3,1	0,46	4,04	1,55	15,28	3,56
2009/01C	Kyparissia	31200	33600	1,17	1,9	10300	0,4	49	2,1	121	113	0,14	2,76	464	30,4	56	20	3,6	0,442	0,5	2,9	0,43	3,61	1,55	14,96	3,39
pumice from excavations																										
L-2	Tell-el Dab^{c}a	30500	30600	1,96	3,0	12000	1,6	51	1,9	111	119	0,14	2,38	420	29,3	52	18	3,7	0,465	0,5	2,7	0,40	3,29	1,44	12,84	3,07
K 13	Tell-el Dab^{c}a	33000	39600	1,37	1,1	11300	0,8	48	n.d.	118	119	0,13	2,60	478	30,1	53	18	3,7	0,470	n.d.	2,9	0,46	3,60	1,51	14,58	3,17
A-5	Tell-el Dab^{c}a	32200	35000	1,79	1,7	12600	1,1	75	n.d.	123	125	0,14	2,71	484	31,2	55	20	3,3	0,493	n.d.	3,0	0,47	3,67	1,54	14,70	3,34
2006/17A	Tell-el Dab^{c}a	33400	37000	1,20	1,9	11100	0,4	53	1,8	122	104	0,15	2,79	502	31,3	57	21	3,8	0,476	0,5	3,0	0,49	3,67	1,59	15,12	3,41
2006/17B	Tell-el Dab^{c}a	33200	37000	1,70	2,5	12600	0,9	55	2,1	120	112	0,49	2,75	498	31,5	57	20	3,8	0,507	0,5	3,0	0,49	3,69	1,58	14,88	3,56
2008/15B	Tell-el Dab^{c}a	30400	36300	1,62	4,4	11500	0,9	51	2,0	119	117	0,15	2,73	462	30,5	54	19	3,6	0,467	0,5	2,9	0,42	3,63	1,53	14,56	3,54
2008/15C	Tell-el Dab^{c}a	32700	39100	1,66	4,5	11700	1,0	51	1,9	115	119	0,16	2,62	451	31,8	53	18	3,7	0,492	0,5	2,9	0,42	3,55	1,50	14,10	3,39
2009/03D	Tell-el Dab^{c}a	33100	36700	1,67	2,8	19200	1,3	65	1,9	119	118	0,14	2,79	472	32,3	58	20	3,6	0,495	0,5	3,0	0,42	3,70	1,53	14,98	3,68
2009/03E	Tell-el Dab^{c}a	31600	35200	1,83	3,5	11700	1,2	51	1,8	115	121	0,15	2,65	476	30,3	55	18	3,6	0,503	0,5	2,9	0,40	3,50	1,52	14,28	3,33
E 80	Ashkelon	31400	41500	2,09	5,8	13200	1,7	58	3,2	124	118	0,20	2,64	466	32,3	56	20	4,0	0,511	0,6	3,0	0,52	3,96	1,71	14,43	3,39
E 146	Ashkelon	35100	38200	1,18	2,1	11600	0,7	60	2,1	127	138	0,16	2,65	483	32,3	58	19	4,0	0,486	0,6	3,0	0,48	3,86	1,62	14,64	3,17
E 174	Ashkelon	34600	35300	1,04	1,4	11000	0,4	55	1,9	123	140	0,13	2,57	472	32,4	55	19	3,9	0,497	0,6	2,9	0,51	3,82	1,56	13,98	3,25
literature values																										
Bathrellos et al.		30200	32800	n.d.	n.d.	11800	1,5	49	n.d.	107,5	122	n.d.	2,6	455	30,7	57	19,6	3,5	0,5	0,6	3	0,5	4	1,5	14	3,2
Upper Crust		28900	28000	11	35	35000	10	71	1,5	112	190	0,2	3,7	550	30	64	26	4,5	0,88	0,64	2,2	0,32	5,8	2,2	10,7	2,8

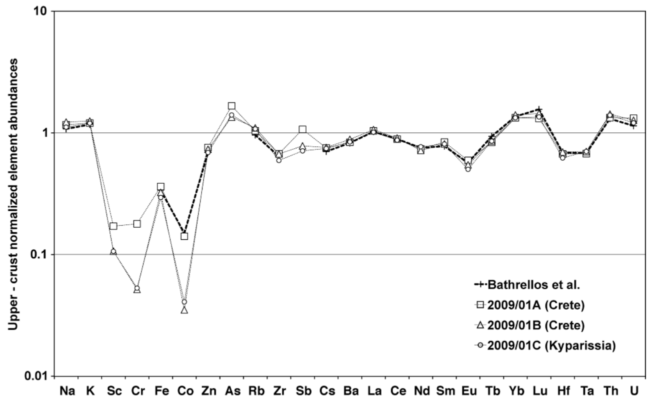

Fig. 3. Upper-crust normalized element abundance patterns of alluvial pumice samples from the Gulf of Kyparissia and Crete, comparing data obtained by INAA (this work) and other methods (BATHRELLOS *et al.* 2009). Normalization factors are taken from TAYLOR & MCLENNAN (1985).

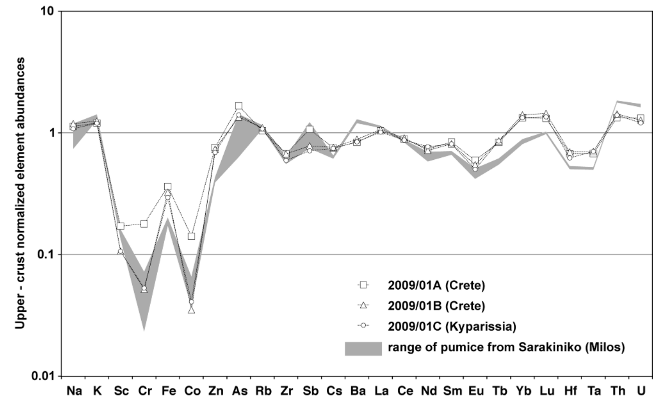

Fig. 5. Upper-crust normalized element abundance patterns of alluvial pumice samples from the Gulf of Kyparissia and Crete in comparison to pumice from Sarakiniko beach, Milos. The grey shaded area shows the range of values for 8 samples from this outcrop (PELTZ *et al.*, 1999). Normalization factors are taken from TAYLOR & MCLENNAN (1985).

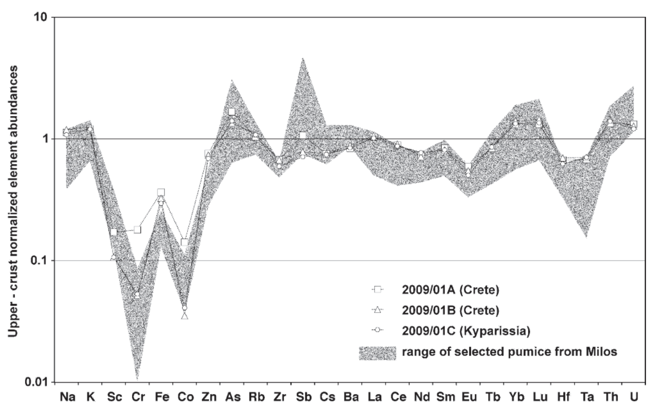

Fig. 4. Upper-crust normalized element abundance patterns of alluvial pumice samples from the Gulf of Kyparissia and Crete in comparison to pumice from Milos. The grey shaded area shows the range of values derived from the data set compiled in PELTZ *et al*. (1999). Normalization factors are taken from TAYLOR & MCLENNAN (1985).

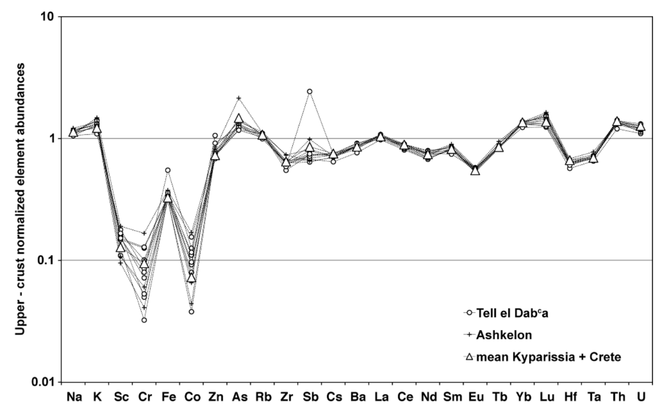

Fig. 6. Upper-crust normalized element abundance patterns of pumice from archaeological excavations compared to the mean values in the samples from Kyparissia and Crete. Normalization factors are taken from TAYLOR & MCLENNAN (1985).

in archaeological samples (e.g. STERBA *et al.,* 2009; BICHLER *et al.,* 2003). The larger scattering for Sc, Cr, Fe and Co in the archaeological samples is more likely to be related to the small size of the archaeological samples. Inhomogeneous distribution of crystalline particles with higher concentrations in these elements like ore minerals, pyroxenes or amphiboles could lead to the aberration from the bulk composition of a representative sample. This problem has been investigated in earlier studies (SCHMID *et al.,* 2000, SAMINGER *et al*., 2000) by separation and analysis of the glass matrix. However, absorption from natural weathering solutions could easily increase the very low concentrations of these elements in the pumice, too. This agrees also with the high absorption potential of pumiceous material, observed in an earlier study (STEINHAUSER & BICHLER, 2008). From the abundances of the elements less affected by this problem, it can be concluded that the source volcano of the investigated archaeological samples from Egypt and the Levant is identical with that of the alluvial pumice collected from Kyparissia and Crete.

Further research should be carried out in order to locate the true source volcano which could lead to a chronological use e.g. by radiocarbon dating of organic material underlying a primary deposit.

ACKNOWLEDGEMENTS

This work is in part supported financially by SCIEM2000, a Special Research Program of the Austrian Academy of Sciences and the Austrian Science Fund. The authors wish to thank in particular the directors of the excavations at Ashkelon and Tell-el-Dab^{c}a, Lawrence E. Stager (Harvard Semitic Museum) and Manfred Bietak (Vienna University) for providing their pumice finds for this research project.

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