ISSN: 0514-7336
DOI: http://dx.doi.org/10.14201/zephyrus201882149163

NEW TECHNOLOGICAL CONTRIBUTIONS TO ROMAN *GARUM* ELABORATION FROM CHEMICAL ANALYSIS OF ARCHAEOLOGICAL FISH REMAINS FROM THE '*GARUM* SHOP' AT POMPEII (I.12.8)

Nuevas contribuciones tecnológicas al estudio de la salsa garum *a partir del análisis químico de restos ictiológicos de la 'Tienda del* Garum*' de Pompeya (I.12.8)*

Álvaro Rodríguez-Alcántara*, Ana M. Roldán-Gómez*, Darío Bernal-Casasola**, Enrique García-Vargas*** and Víctor M. Palacios-Macías*

** Dpt. of Chemical Engineering and Food Technology. Faculty of Sciences-UCA. República Saharahui Avenue, 9. 11510 Puerto Real (Cádiz). E-mail: alvaro.rodriguezalcantara@uca.es; ana.roldan@uca.es; victor.palacios@uca.es. ORCID ID: 0000-0003-1608-8708; 0000-0003-1608-8708; 0000-0002-6225-0975*
*** Dpt. of History, Geography and Philosophy. Faculty of Philosophy and Letters-UCA. Dr. Gómez Ulla Avenue, 1. 11003 Cádiz. E-mail: dario.bernal@uca.es. ORCID ID: 0000-003-1107-5318*
**** Dpt. of Prehistory and Archaeology. Faculty of Geography and History-US. Doña María de Padilla Street, s/n. 41004 Sevilla. E-mail: egarcia@us.es. ORCID ID: 0000-0002-8361-1071*

Recepción: 27/2/2018; Revisión: 21/07/2018; Aceptación: 16/09/2018

Abstract: The *garum* fish sauce was a popular ingredient in Roman gastronomy; however, its production process is still unknown, and few remains have been found in the whole ancient world. The aim of this study was to implement a technical approach to determine the ancient *garum* preparation methods, ingredients and additives by comparing the chemical compositions of archaeological fish remains found in six vessels –*dolia*– at the '*Garum* Shop' of Pompeii (Italy) and *garum* obtained from the Gargilio Martial's classical recipe. Some organic and inorganic parameters and compounds specific for fish sauces with great physicochemical stability were determined either in the *dolia* but also in the experimental reconstruction. The total N, fat, ash content, fatty acid, and mineral composition were determined. Differences found in the N and fat contents of *dolia* could indicate that different *garum* batches were being manufactured at the moment of the Vesuvian eruption in the '*Garum* Shop'. Fatty acid results showed a similar profile for the remnants and reference *garum*. The mineral profile, Ca/P ratio and Fe contents reported could be explained by the use of lime in the *garum* preparation or sanitizing stage, and food coloring respectively.

Key words: *dolia*; fatty acids; minerals; Experimental Archaeology; Roman fish sauces.

Resumen: La salsa *garum* fue un ingrediente popular en la gastronomía romana, sin embargo, su proceso de producción sigue siendo desconocido y se han conservado pocos restos del producto en el mundo antiguo. El objetivo del estudio fue realizar una comparativa de la composición química de los restos de seis *dolia* procedentes de la 'Tienda del Garum' de Pompeya (Italia) y del *garum* obtenido a partir de la receta clásica de

Gargilio Marcial, para determinar técnicas de elaboración, ingredientes y aditivos. Se determinaron algunos indicadores orgánicos e inorgánicos específicos para las salsas de pescado con gran estabilidad fisicoquímica. Se han determinado los contenidos en nitrógeno total, grasas, cenizas, ácidos grasos y perfil mineral tanto en las muestras arqueológicas como en las muestras obtenidas por arqueología experimental. Las diferencias observadas en los contenidos en nitrógeno y grasas entre los *dolia* podrían indicar que en la 'Tienda del *Garum'* se estaban elaborando diferentes lotes de salsa de pescado. Los resultados observados en el perfil de ácidos grasos muestran similitudes entre los restos arqueológicos y el *garum* reproducido. El perfil mineral, la relación Ca/P y los niveles de hierro observados podrían explicarse por el uso de yeso, como aditivo, como agente desinfectante o como colorante.

Palabras clave: *dolia*; ácidos grasos; minerales; Arqueología Experimental; salsas de pescado romanas.

1. Introduction[1]

Garum was a liquid produced by the hydrolysis of small whole fish in the presence of salt, aromatic herbs and spices, through natural fermentation over several months (Curtis, 1991; Curtis, 2009). *Garum* sauce was the most popular condiment in Roman gastronomy, but there is not much information about *garum* production methods except that two fractions were obtained: a liquid called *garum* or *liquamen* –terms closely related and used by the Romans as synonymous– and a solid fraction containing non-solubilized remains, called *allec*, resulting from the filtration of the *garum* (Curtis, 1991, 2009; Desse-Berset and Desse, 2000; García *et al.*, 2014). The importance of these fish sauces in daily Roman life, as well as its high impact in Antiquity have been treated in detail by many authors, from the social to the economical point of view (Étienne and Mayet, 2002); and from the Strait of Gibraltar (Bernal, 2011) or Sicily (Botte, 2009) to the Black Sea (Bekker-Nielsen, 2005).

Literary sources provide few details about the preparation of the sauce, although it is known that it was produced at an artisanal scale using large storage jars –*dolia*– or other ceramic containers with different capacities and at an industrial scale in fish-salting factories all around the Mediterranean Sea and the Atlantic Ocean (Ponsich and Tarradell, 1965; Desse-Berset and Desse, 2000; Étienne and Mayet, 2002; Reese, 2002). It is also known that different specialty types of *garum* were developed that were highly prized in the Roman world, such as the *garum haimation* or the 'blood *garum*' (Van Neer and Parker, 2008). This product was very common in Roman fish-salting factories located around the migration route of Bluefin tuna –*Thunnus thynnus*–, where in addition to small fish, the blood, innards or gills of tuna were used, all of which were by-products generated during the *ronqueo* or cutting process (García and Bernal, 2009; Curtis, 2009)[2].

The hypothetical *garum* production process could be similar to modern fish sauce production processes carried out in Asia (Palacios *et al.*, 2016) and Italy (Carannante, 2010). Currently, Asian sauces such as *nouc-mam* –Vietnam– or *nam-pla* –Thailand– are made with similar ingredients, and some authors have remarked that modern Asian fish sauces and ancient Roman preparations likely used the same technological resources for manufacture (Curtis, 2009; Grimal and Monod, 1952; Ruddle and Naomichi, 2010; Grainger, 2011; Smriga *et al.*, 2010), and it is possible to use the extensive studies on Asian fish sauces to reconstruct the process of *garum* sauce production providing contemporary reference materials for comparative analyses. Some authors have used modern products and contemporary preparations as a reference in their studies on archaeological remains. Dallongeville *et al.*, (2011) have characterized ceramics imbued with

[1] This work is based on research projects supported by the Spanish Ministry of Economy and Competitiveness/FEDER EC (refs. no. HAR 2013-43599P and HAR2016-78691-P).

[2] Rodríguez, A.; Roldán, A.; Bernal, D.; García Vargas, E. and Palacios, V.: "Analysis of the productive capacity of the 'Garum Shop' (Pompeii I.12.8) using experimental archaeology methods". In *Conference Proceedings of the Congress Fesciti Cretaria (2016)*. Pompeii (Italy), in press.

fresh anchovies as a reference for archaeological samples and to optimize the methods of protein extraction from remains. Pecci *et al*. (2013b) used GC/MS to identify organic acids in residues of wine using fresh wine in ceramic vessels as a reference. Another study, performed by Garnier *et al*. (2003) on the archaeological remains of food, studied the reproduction of ancient techniques and the simulation of the effects of time on contemporary samples. These authors used thermal-assisted hydrolysis and methylation on modern products to provide reference analytical data for the comparative study of archaeological materials.

Many new techniques and methods, such as biochemical, DNA, zoological or botanical analyses, are actually applied in archaeology and contribute with valuable information about the chemical and physical characteristics of archeological remnants (Brown and Brown, 1992, 2011). Archaeological sources provide very valuable materials that deal with the use of raw materials in ancient times and can make a crucial contribution to the determination of the

FIG. 1. *A) Photograph of the 5 dolia (D1-D5 from left to right) analysed at the central part of the* Garum *Shop (Pompeii, Italy) (photo by Univ. of Cádiz). Dolium D6 was in the central part of the garden of 'Ambiente 9'; B) detail of the fish vertebrae inside the ceramic vessels (photo by C. G. Rodríguez-Santana and J. Morales).*

ingredients and additives used during production. A practical example of this are the archaeological research on the six *dolia* found in the '*Garum* Shop' of Pompeii (Curtis, 1979; Curtis, 1983) conducted by a Spanish-Italian group (Bernal and Cottica, 2010; Bernal *et al.*, 2013), which has allowed new interdisciplinary studies on experimental archaeology of small-scale *garum* preparations (García *et al.*, 2014).

For the archaeological remains of fish or fish sauces in ceramic vessels, studies have focused on organic compounds as fatty acids, sterols and acylglycerol, due to the great physicochemical stability of these compounds (Brown and Heron, 2004; Garnier *et al.*, 2009; Evershed, 1993; Dallongeville *et al.*, 2011; Garnier *et al.*, 2003; Passi *et al.*, 2015; Pecci *et al.*, 2013a; Regert, 2011), that could serve as biomarkers for the identification of ingredients that cannot be detected by bone or pollen analysis (Evershed, 2008). Fatty acids detection by GC-MS techniques was a common method to determine the molecular composition and the isotopic characteristics in archaeological remnants (Regert, 2011).

Despite the real fact of considering some fatty acids as biomarkers for animal fats or other organic residues, determining the nature and origin was a great challenge (Regert, 2011). The application of new techniques as proteomic or GC-MS combined with carbon isotopic analysis opened new possibilities for the identification of protein species in ancient adherences (Dallongeville *et al.*, 2011).

Fish sauces are a notable source of nitrogen and other nitrogen based compounds as proteins and free amino acids, especially glutamic acid, which are detected in the modern fish sauces of Italy and Asia (Ikeda, 2002; Beddows, 1998; Smriga *et al.*, 2010). These compounds have been detected at very low levels in the remains of *garum* contained in two vessels of the '*Garum* Shop' at Pompeii (Smriga *et al.*, 2010). Thus, a pattern of free amino acids, and especially glutamate, could be used as a reference to identify fish sauce remains in archeological samples (Smriga *et al.*, 2010). Therefore Nitrogen measurements could be used to calculate the organic matter present in samples and as indicative of degradation level due to time; however the Kjeldahl method only allows to know the total soluble nitrogen from all the sources, organic –aminoacids, nucleics acids– and inorganic –ammonia compounds and others–. Mineral profiles are important components in fish sauces as well, and they have also been the object of many studies and analyses (Mika *et al.*, 2016; Mizutani *et al.*, 1992). However, there are few studies linking the mineral profile with the identification of ingredients in archaeological remains. Calcium and phosphorus can be reference characteristic elements of the composition of the bone remains of fish (Toppe *et al.*, 2007), and the presence of sodium could identify its use both in salted fish and fish sauces. However, the sodium levels in *garum* reported by Smriga *et al.* (2010) were approximately 20 million times lower than those in contemporary fish sauces (Crisa and Sands, 1975), possibly due to the post-excavation period and occasional rain that partly washed out its contents (Smriga *et al.*, 2010).

The objective of this study was to implement a technological approach to the production methods and possible ingredients or additives used in ancient *garum* production based on analysis of the proximal composition, fatty acids and minerals of the remains of solid *garum* found in six *dolia* of the *Bottega del Garum* (Pompeii, Italy) and the by-products obtained in the *garum* reconstruction process, which was elaborated starting from the recipe, the primary components identified in these *dolia*, and the Asian sauce production process.

2. Materials and methods

2.1. Samples of the archaeological remains

Samples of the archaeological remains of *garum* –*allec*– were extracted from a depth of 10 cm from six *dolia* –D1-D6– preserved in the so called 'Ambiente 9' in the '*Garum* Shop' of Pompeii –I.12.8– (Fig. 1A). Samples were found in an extraordinary state of preservation (Fig. 1B), the six vessel appeared without lapilli or volcanic ashes (Curtis, 1979; Bernal *et al.*, 2013). Previous zoological analysis of remnants of these *dolia* showed anchovy

(*Engraulis encrasicolus*; Fig. 1B) and pollen from culinary spices (Curtis, 1979; Bernal *et al.*, 2013). These remnants were characterized as the last *garum* productions before the Vesuvian eruption (Bernal *et al.*, 2013; Carannante, 2010; García *et al.*, 2014; Curtis, 1979) and were found in the solid form, corresponding to *allec*.

2.2. Allec reference samples

Contemporary samples of reference *allec* were obtained from modern *garum* elaborations following the reconstruction process based on Gargilio Martial's classical recipe 'Confetio Gari' (García *et al.*, 2014). Different *garum* batches were prepared using anchovy –80%–, salt –15%– and Mediterranean spices –5%–, placed in layers –spices-fish-salt– inside a 5 L thermostated glass vessel. The temperature was fixed at 50 ± 5 ºC to maximize the fish protease activity, and the vessels were sealed for a week and maintained in the dark to simulate an amphorae exposed to the sun. Starting on day 8, the mixture was stirred for 5 min/day for 7 more days (García *et al.*, 2014). At these times, fractions of *liquamen* and *allec* were separated by filtering through linen, and the *allec* fraction –*allec* reference– was collected –5 kg– and kept deep-frozen until analysis. Prior to analysis, all the samples were lyophilized and homogenized using a mill and an agate mortar.

2.3. Fresh anchovy samples

The anchovy samples were acquired at a local market in Cadiz. Five kg of fresh fish were deep-frozen in one piece without beheading or eviscerating, and the rest of fish was used for *garum* elaboration. All samples were stored under deep-freeze (-80 °C) until the time of analysis. Prior to analysis, all the samples were lyophilized and homogenized using a mill and an agate mortar.

2.4. Analytical determinations

2.4.1. Moisture

Water lost in each sample was gravimetrically determined using a RADWAG PS 360/C/1 scale –RADWAG, Poland– by measuring the decrease in total mass of the fresh matter and the final weight after kiln-drying in a drying oven P-Selecta Digitronic-TFT –Selecta Group, Spain– at 95 ºC +/- 5 ºC. Final values were accepted after three weighings without significant changes –± 0.001 g–.

2.4.2. Total N contents

The total N contents were determined using the Kjeldahl method (AOAC, 2000). Samples –1 g– were digested in an automated digester, model DK6 –Velp Scientific, Italy–, and distilled by steam distillation equipment, model UDK127 –Velp Scientific, Italy–. Nitrogen measurements were used to calculate the organic matter present in samples and as indicative of degradation level due to time. However the Kjeldahl method only allows to know the total soluble nitrogen from all the sources –aminoacids, nucleics acids, ammonia compounds and others–.

2.4.3. Fat and fatty acid content

Fat content was determined by the Soxhlet method (AOAC, 2000) using an automated Solvent Extractor SER148 –Velp Scientifica, Italy– and n-hexane as the extraction solvent. Total fat content was calculated as the difference between the dry sample weight –1g– before and after the fat extraction. The hexane-fat mix was evaporated using a rotary evaporator Hei-VAP Value G1 –Heidolph, Germany– at 60 °C until total hexane evaporation. The fat collected was stored at 4 °C for methylation and later fatty acid determination.

Fatty acids were determined by gas chromatography after derivatization to methyl esters (FAMEs) according to the IUPAC standard method. Analysis of the FAMEs (Dieffenbacher and Pocklington, 1992) was performed on an Agilent 7890-A series gas chromatography system –Agilent Technologies, Germany– coupled to a triple-quadrupole cut-off high-resolution mass spectrometer, model Synapt G2-S –Waters, USA–, equipped with a DB-5 capillary column (30 m x 0.25 µm x DFID 0.25 mm). The column temperature was programmed from 190 °C to 250 °C at 2 °C/min. Methyl cis-10-heptadecenoate

–Sigma-Aldrich, USA– (C17:0) was used as internal standard. Reference fatty acid methyl esters –FAMEs– of marine source were used to identify and quantify FAMEs –Sigma-Aldrich, USA–, and PUFA no. 1 –Marine Source, Sigma-Aldrich, USA– was used as a standard reference.

2.4.4. Ash and mineral content

Ash content was determined by the incineration of 1 g of dried sample in a muffle oven at 500 °C for 2 h. Ashes, containing the inorganic fraction, were dissolved in nitric acid (AFNOR, 1996) for mineral extraction: Phosphorous (P), Calcium (Ca), Sodium (Na), Magnesium (Mg), Potassium (K), Iron (Fe), Copper (Cu) and Zinc (Zn). All the minerals were determined by inductively coupled plasma atomic emission spectroscopy –Iris Intrepid ICP-AES, Thermo Scientific, USA–.

2.5. Color analysis

Color measurements in the CIELAB space were obtained with a portable CM-2600d Spectrophotometer –Konica Minolta Optics, Inc., Japan–. The CIELAB space –CIE, 1978– is represented by lightness –L*–, redness –a*– and yellowness –b*–, which represent the balance between black –L*= 0– and white –L*= 100–, red –a*> 0– and green –a*< 0–, and yellow –b*> 0– and blue –b*< 0–, respectively. The instrument was calibrated with a white ceramic tile before each series of measurements. All samples were measured three times at three different points using illuminant D65 and 10° observer settings.

2.6. Statistical analysis

All results are expressed as the mean values ± standard deviation –SD–. The data were subjected to one-way analysis of variance –ANOVA–. Differences were accepted as statistically significant at a probability of $p < 0.05$.

Principal components analysis (PCA) with orthogonal rotation –'varimax'– as the factor extraction method was applied to analytical data of *dolia* remains to find patterns in the samples. Correlations were accepted as statistically significant at a probability of $p < 0.05$. Statistical evaluations and PCA analysis were carried out with the IBM SPSS Statistics 22 software –IBM Corporation, USA–.

3. Results and Discussion

3.1. Chemical composition

Figs. 2 and 3 shows the results corresponding to the chemical composition of the archaeological remains –*garum*– with the reference of *allec* and fresh anchovy samples. The *garum* remains showed a high degree of dehydration, with a moisture value < 0.3%. The ash levels fluctuated between 92-98%, resulting in a calculated content of organic matter between 2 and 8%. These results suggest a good state of sample conservation considering that they are 2.000 years old. Some authors (Luongo *et al.*, 2003; Gurioli *et al.*, 2005) reported that during the 79 AD Mt. Vesuvius eruption, the *dolia* were covered and buried under 1-3 meters of volcanic ash. This circumstance, and the *dolia* placement

	D1	D2	D3	D4	D5	D6	*allec* reference	fresh anchovy
Moisture (%)	0.2 ± 0.1*	0.3 ± 0.1*	0.2 ± 0.1*	0.1 ± 0.1*	0.2 ± 0.1*	0.3 ± 0.1*	56.2 ± 1.3	72.3 ± 4.1
Ash (% DW)	92.1 ± 1.3*	93.3 ± 1.2*	96.9 ± 1.5*	98.6 ± 2.3*	98.2 ± 1.2*	97.7 ± 0.9*	50.7 ± 1.8	32.2 ± 2.0
Fats (% DW)	2.33 ± 0.11*	2.09 ± 0.08*	1.14 ± 0.12*	0.50 ± 0.04*	0.79 ± 0.07*	0.99 ± 0.06*	10.4 ± 1.3	7.62 ± 1.12
Total N (% DW)	0.61± 0.06*	0.45 ± 0.07*	0.26 ± 0.03*	0.08 ± 0.01*	0.17 ± 0.02*	0.19 ± 0.03*	3.36 ± 0.53	7.84 ± 1.22
Fill level (cm)	20.0 ± 1.3*	20.0 ± 1.1*	20.0 ± 1.1*	35.0 ± 1.7*	5.0 ± 0.7*	20.0 ± 1.2*	-	-

Results are the means ± SD (n = 3); nd = not detectable.
* Significantly different from the values detected in *allec* reference ($p < 0.05$) using ANOVA

FIG. 2. *Chemical composition of* dolia*, allec reference and fresh anchovy samples.*

between the *garum* shop walls, could have preserved these samples from the pyroclastic current. Therefore, the temperature inside the *dolia* could have been maintained under 100 °C, limiting the combustion of organic matter.

As shown in Fig. 2, significant percentages of fat were observed, ranging between 0.5 and 2.33% of the dry weight. These results corroborate the good state of conservation for the archaeological samples and the great physico-chemical and microbiological stability of this group of substances (Evershed, 1993, 2008). D1 and D2 showed the highest fat levels –2%–, corresponding to approximately 23% of the fat in the *allec* reference samples –10.4%–, while D4 and D5 showed the lowest percentages –0.50 and 0.79%, respectively–. Assuming that a faithful *garum* preparation process was used for the reconstruction, these results make it possible to estimate that the degradation level for fats is in the range of approximately 77-95% over 2000 years since preparation.

As expected, the nitrogen percentages in the samples were relatively low –0.08-0.61% dry weight– and showed a linear correlation with the fat contents –r^2 = 0.94–. The linear correlations of total N and fat with ash are r^2 = -0.94 and -0.95, respectively. So, these results showed that nitrogen present in the archaeological remains was organic nitrogen, and its degradation level was very similar to that of fat –90%–. D1 and D2 showed higher total N levels –0.61 and 0.45%–, and the lowest level corresponded to D4 –0.08%–, D5 –0.17%– and D6 –0.19%–. It is probable that the organic nitrogen in the samples belongs to amino-acids and other nitrogen

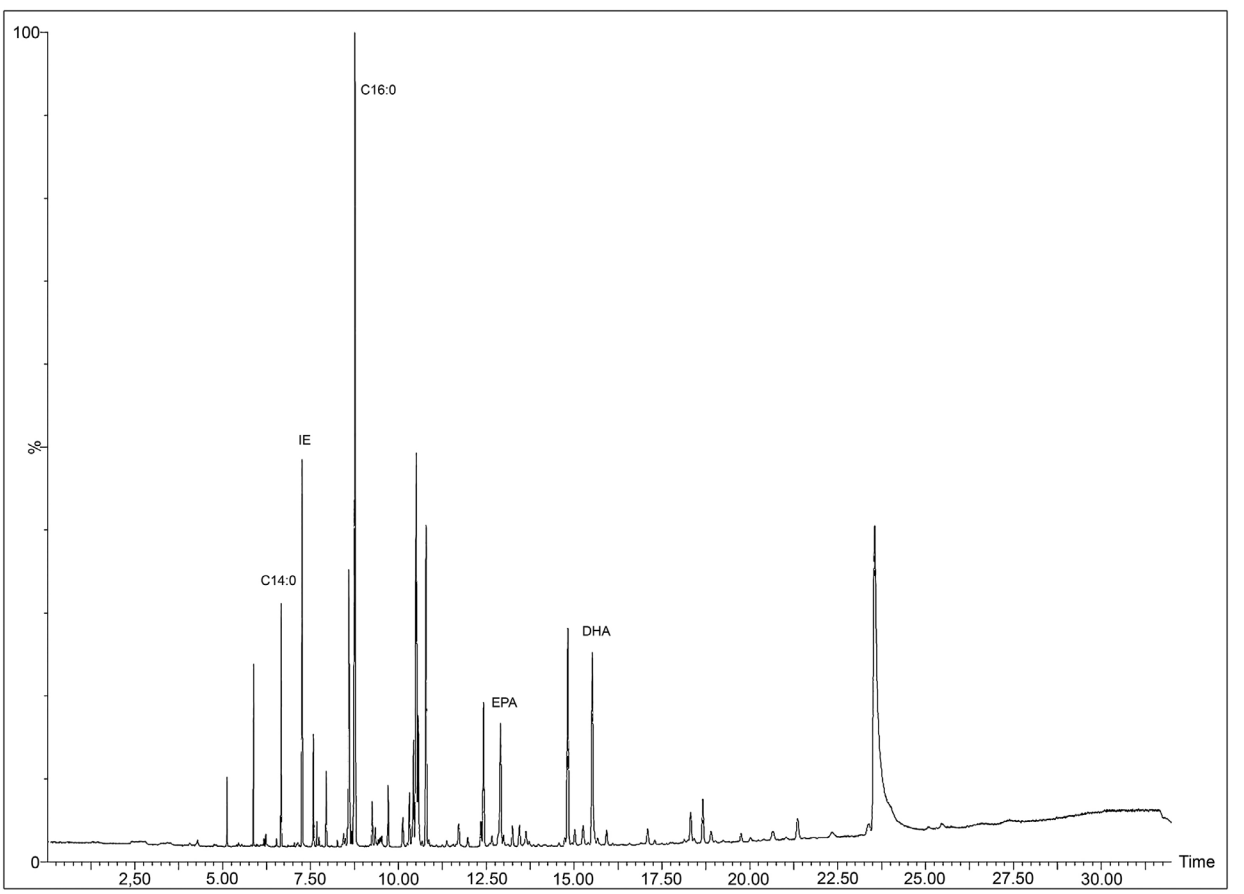

Fig. 3. *Representative chromatogram showing principal fatty acids identified in the archaeological samples (IE = internal standard; EPA = eicosapentaenoic acid; DHA = docosahexaenoic acid).*

	D1	D2	D3	D4	D5	D6	Allec reference	Fresh anchovy
C13:0	0.20±0.02	0.04±0.01	0.02±0.01	n/d	0.01±0.00	0.01±0.00	0.01±0.00	0.01±0.00
C14:0	3.01±0.13*	2.03±0.23*	0.57±0.08*	0.11±0.02*	0.15±0.03*	0.20±0.02*	5.18±1.10	1,23±0.04
C15:0	0.25±0.01*	0.14±0.01*	0.10±0.01*	0.02±0.00*	0.10±0.01*	0.05±0.01*	0.66±0.02	0.43±0.01
C16:0	5.98±0.21*	7.26±0.36*	4.39±0.45*	0.07±0.02*	0.06±0.01*	2.17±0.10*	20.20±1.22	9.30±0.82*
C18:0	n/d	n/d	2.00±0.07*	n/d	n/d	0.72±0.04*	4.17±0.29	1.78±0.06*
C20:0	n/d	n/d	n/d	n/d	n/d	n/d	0.16±0.01	0.10±0.01
C22:0	0.42±0.03	n/d	0.35±0.02	0.02±0.00*	0.04±0.01*	0.09±0.01*	0.35±0.03	0.20±0.01
∑SFAs	9.85±0.40*	9.47±0.61*	7.43±0.64*	0.22±0.04*	0.37±0.06	3.24±0.18*	30.74±2.67	13.06±0.95
C16:1	0.64±0.12*	n/d	0.38±0.04*	0.09±0.01*	0.11±0.02*	0.27±0.05*	2.06±0.23	1.08±0.18*
C18:1 n-9	0.02±0.00*	n/d	0.29±0.05*	n/d	0.08±0.01*	0.21±0.02*	5.04±0.32	1.66±0.17*
C20:1 n-9	2.15±0.21	3.00±0.19	n/d	n/d	0.02±0.00*	0.05±0.01*	1.62±0.09	0.49±0.08*
C24:1 n-9	1.32±0.05*	1.36±0.08*	0.01±0.00*	n/d	0.01±0.00*	n/d	0.64±0.03	0.44±0.08*
∑MUFAs	4.12±0.38*	4.36±0.27*	0.68±0.09*	0.09±0.01*	0.22±0.03	0.54±0.08	9.37±0.67	3.67±0.51
C18:2 n-9	n/d	n/d	0.01±0.00*	n/d	0.09±0.01*	0.09±0.02*	0.75±0.16	1.07±0.09
C18:3 n-3	n/d	n/d	0.02±0.01*	n/d	0.09±0.02*	0.09±0.01*	0.75±0.18	0.99±0.03
C20:4 n-6	0.43±0.05	0.13±0.02	0.02±0.01*	0.01±0.00*	0.01±0.00*	0.02±0.01*	0.34±0.05*	0.92±0.07*
C20:5 n3	2.21±0.13	2.93±0.10	0.96±0.07*	0.06±0.02*	0.07±0.01*	0.83±0.06*	2.73±0.09	4.73±0.15*
C22:5	0.38±0.05	0.41±0.03	0.03±0.01*	0.01±0.00*	0.01±0.00*	0.02±0.00*	0.51±0.08	0.26±0.06*
C22:6 n-3	3.54±0.12*	5.59±0.21	1.59±0.07*	0.08±0.02*	0.11±0.02*	0.98±0.05*	6.98±0.28	12.31±0.83*
∑PUFAs	6.56±0.35*	9.05±0.36*	2.63±0.17*	0.16±0.04*	0.38±0.06	2.01±0.15*	12.05±0.84	20.10±1.23
(∑SFAs+∑MUFAs)/∑PUFAs	2.12	1.52	3.08	1.93	1.55	1.88	3.32	0.83
∑mg/100 g DW	20.53±1.13	22.89±1.24	10.74±0.90	0.48±0.09	0.96±0.15	5.79±0.41	52.16±4.18	36.83±2.69

SFAS = saturated fatty acids; MUFAS = monounsaturated fatty acids; PUFAS = polyunsaturated fatty acids.
Results are the means ± SD (n = 3); nd: not detectable.
* Significantly different from the values detected in *allec* reference ($p < 0.05$) using ANOVA.

	D1	D2	D3	D4	D5	D6	*allec* reference	fresh anchovy
c13:0	0.95	0.18	0.14	0.81	1.04	0.09	0.03	0.04
c14:0	14.65	8.86	5.30	23.30	15.28	3.40	9.94	3.35
c15:0	1.19	0.61	0.92	4.34	10.69	0.79	1.26	1.17
c16:0	29.11	31.73	40.89	13.67	6.70	37.56	38.73	25.25
c18:0	-	-	18.64	-	-	12.51	7.99	4.83
c20:0	-	-	-	-	-	-	0.31	0.27
c22:0	2.06	-	3.29	4.48	4.39	1.57	0.68	0.54
∑SFAS	**47.97**	**41.37**	**69.18**	**46.61**	**38.11**	**55.93**	**58.94**	**35.45**
c16:1	3.12	-	3.57	19.75	11.28	4.73	3.95	2.94
c18:1 n-9	0.08	-	2.69	-	8.51	3.30	9.67	4.50
c20:1 n-9	10.46	13.12	-	-	1.85	0.86	3.11	1.32
c24:1 n-9	6.41	5.95	0.08	-	1.13	-	1.23	1.20
∑MUFAS	**20.08**	**19.07**	**6.34**	**19.76**	**22.77**	**9.30**	**17.96**	**9.96**
c18:2 n-6	-	-	0.11	-	9.28	1.47	1.44	2.91
c18:3 n-3	-	-	0.18	-	9.28	1.47	1.44	2.68
c20:4 n-6	2.11	0.57	0.16	1.94	0.94	0.33	0.65	2.51
c20:5 n-3	10.75	12.79	8.98	12.42	7.18	14.27	5.23	12.86
c22:5	1.86	1.79	0.27	1.99	0.88	0.31	0.97	0.70
c22:6 n-3	17.22	24.41	14.78	17.28	11.55	16.92	13.37	32.93
∑PUFAS	**31.95**	**39.55**	**24.48**	**33.64**	**39.12**	**34.77**	**23.10**	**54.59**

FIG. 4. *A) Fatty acid content (mg/100 g of DW sample) of* dolia, *allec reference and fresh anchovy samples; B) fatty acid profile (%) of* dolia, allec *reference and fresh anchovy samples.*

compounds from the fish sauce composition. In fact, Smriga *et al.* (2010) have reported amino acid in the remains of D6.

Therefore, *garum* based on the chemical composition results obtained, it is possible to determine different clusters of *dolia* in the '*Garum* shop': the first one formed by D1 and D2 that is relatively high in fat and total N and low in ash; the second formed by D3 and D6 with medium percentages; and a third group –D4 and D5– with very low total N and fat levels and high ash levels.

According to the different stages of the *garum* production process, these results could indicate that different *garum* batches were being manufactured in the '*Garum* Shop' at the moment of the Vesuvian eruption, and/or these batches could be in different stages of the production process. At the final stages of *garum* production –modern and ancient–, two products are obtained, a liquid phase –named *liquamen* or *garum*– and a solid phase –*allec*–, which must be separated by filtering in linen (Curtis, 1991; Bernal and Sáez, 2006). As shown in Fig. 2, a decrease in the total nitrogen of the *allec* reference with respect to the fresh anchovy occurred. This can be explained because when the *allec* and *garum* are separated, part of the total nitrogen passes to the liquid phase –*garum*–, decreasing its content in the *allec*. Therefore, the low total nitrogen content in D4 and D5 with respect to other *dolia* could indicate that these remains corresponded with the *allec* after the filtering process. These results could indicate that D4 and D5 belong to the same batch, although D5 comes from a vessel with low levels of remains –approximately 5 cm– (Fig. 2), which could indicate that it was in the stage of removing *allec* or another phase of post-manufacturing.

On the other hand, the compositions found in D3 and D6 suggest that *garum* was being manufactured at the time of the eruption and that in these *dolia*, both *garum* and *allec* were present. The liquid phase inside these *dolia* suffered progressive evaporation through the vessel wall over the years and as a result of the eruption. The D1 and D2 *dolia* (Fig. 2) were in the same process stage; however, the differences observed between these two groups could be attributable to the use of different ingredients in their preparation.

3.2. Fatty acid profile

Figure 4a shows the fatty acid content in the *garum* archaeological remnants, *allec* reference and fresh anchovy. First, an increase in total fatty acid content –41%– in the *allec* reference can be observed with respect to fresh anchovy. The production process of *garum* involves the addition of herbs and spices to the high fatty acid content. However, these ingredients do not contribute qualitatively to the new fatty acids in the fresh anchovy. Therefore, the content of these compounds in *allec* is high, although part of them pass to the liquid phase after *garum* separation. All of the remnants have significant levels of fatty acids, and clusters of *dolia* are also observed. In this way, D1 and D2 show high similar total fatty acid levels of approximately 20-23 mg/100 g dry weight (Fig. 4a). These results for the archaeological samples are 50% of the total fatty acids of the *allec* reference –52.16 mg/100 g dry weight–. D3 and D6 show lower total fatty acids levels than D1 and D2 but are similar to each other, at 10.74 and 5.79 mg/100 g dry weight, respectively. Finally, the fatty acid contents detected in D4 and D5 are the lowest –0.48-0.96 mg/100 g dry weight–, representing only 1-2% of the total fatty acids present in the *allec* reference (Fig. 4a). These results corroborate the clusters observed in the chemical analysis.

On the other hand, the fatty acid profile –%– (Fig. 4b) shows increases in Saturated Fatty Acid –SFA, 23%– and Monounsaturated Fatty Acid –MUFA, 8%– and a decrease in Polyunsaturated Fatty Acid –PUFA, 31%– in the *allec* reference with respect to fresh anchovy. This could be due to the fatty acid hydrolytic process, which is characteristic of anchovy sauces, as shown in previous studies by Cha *et al.* (1985). However, the PUFA content reduction –8.05 mg/100 g– is not the same as the increase in SFA and MUFA content –23.38 mg/100 g– (Fig. 4a), probably due to the minimum contribution of spices to the SFA. The SFA, MUFA and PUFA remains ratio of *garum* is similar to that of the *allec* reference

(Fig. 4b). The SFA are the main fatty acids, with percentages between 38% and 69%, followed by PUFAS –24-39%– and MUFA, with percentages between 6 and 22%. However, fresh anchovy shows higher content of PUFA –54%–, followed by SFA –35%– and finally MUFA –10%–. Similar results have been reported by Zlatanos *et al*. (2007) in Mediterranean Sea anchovies captured at July. Therefore, the ratio of (∑SFAS + ∑MUFAS)/∑PUFAS in the remains and *allec* reference are similar with values higher than 1.

Significant differences are observed in the fatty acid profile between the *allec* reference and archaeological remains. With respect to SFA, the C14:0, C16:0 and C18:0 are the majority in the *allec* reference (Fig. 4a), while in the remains, C18:0 is not detected except for in D3 and D6. Moreover, the MUFAS C16:1, C18:1 n-9, C20:1 n-9 and C24:1 n-9 are principally present in the *allec* reference, while some are not detected in the remains, with the exception of C18:1 n-6 in D3 and D6, and C20:1 and C24:1 in D1 and D2. In these latest *dolia*, the C20:1 and C24:1 content is higher than in the *allec* reference, which is remarkable given the age of the samples. This result could indicate that the *garum –liquamen–* and *allec* was not separated and that additional fresh matter that is rich in these fatty acids was used in the ancient *garum* preparation in these *dolia*. According to sources, these fatty acids are characteristics of fish liver from cod, tuna and other fish (Loftsson, 2016). Regarding the PUFA content (Fig. 4b), the main fatty acids that were found were DHA –C22:6 n-3– and EPA –20:5 n-3–, and the highest levels were found in D1 and D2 at a similar content to that of the *allec* reference, which could also corroborate the use of any additional source rich in these fatty acids in this *dolia* as liver.

3.3. Mineral composition

Figure 5 shows the mineral composition in the archaeological remnants, *allec* reference and fresh anchovy. As can be observed, Ca and P were the main minerals and represented 64 and 31%, respectively, of the total mineral profile in all the archaeological samples, with the exception of D5. The same result is observed in the fresh anchovy; however, the *allec* reference shows Ca and P contents that are lower due to the predominance of sodium, one of the ingredients of *garum* preparation. The Na content in all the remains should be high, but the results show otherwise. Smriga *et al*. (2010) reported similar low Na content in D6 and explained this observation by the effect of rain on the exposed *dolia* during the post-excavation phase.

Some authors (Toppe *et al*., 2007; Martínez-Valverde *et al*., 2000) determined the fish bone composition as an extracellular organic matrix covered in hydroxyapatite [$Ca_5(PO_3)_3OH_2$] based on a specific Ca/P ratio. The Ca/P ratio for all the samples, with the exception of D5, is very similar (medium value = 2), which indicates that the Ca and P contents come basically from the bone. The D5

	D1	D2	D3	D4	D5	D6	*allec* reference	fresh anchovy
P	107.24 ± 12.32*	155.40 ± 9.28*	161.10 ± 7.45*	152.16 ± 4.31*	5.26 ± 2.32*	142.13 ± 10.92*	14.28 ± 1.76	53.64 ± 3.24*
Ca	232.59 ± 25.13*	312.67 ±21.43*	313.46 ± 20.70*	288.41 ± 22.64*	238.35 ± 19.87*	291.52 ± 21.12*	28.77 ± 1.89	96.86 ± 3.95*
Na	6.41 ± 0.87*	5.36 ± 1.16*	3.91 ± 0.48*	4.60 ± 0.34*	1.66 ± 0.17*	3.22 ± 0.11*	144.67 ± 11.77	5.43 ± 1.41*
Mg	6.65 ± 2.09*	7.73 ± 1.88*	1.87 ± 0.23	3.18 ± 0.89*	2.17 ± 0.73	4.16 ± 1.10*	2.16 ± 0.68	2.08 ± 0.33
K	3.26 ± 1.12*	0.95 ± 0.08*	0.99 ± 0.02*	1.14 ± 0.07*	5.67 ± 1.02	0.72 ± 0.04*	6.44 ± 1.17	13.64 ± 2.09*
Fe	4.55 ± 1.72*	4.23 ± 1.69*	3.88 ± 1.77*	4.89 ± 1.38*	7.42 ± 1.92*	3.83 ± 1.07*	0.09 ± 0.01	0.11 ± 0.07
Cu	0.06 ± 0.01	0.06 ± 0.02	0.05 ± 0.01	0.07 ± 0.02	0.05 ± 0.01	0.04 ± 0.01	0.60 ± 0.12	0.02 ± 0.01
Zn	0.48 ± 0.09	1.07 ± 0.19*	0.95 ± 0.07	0.80 ± 0.05	0.06 ± 0.01	0.79 ± 0.07	0.14 ± 0.03	0.14 ± 0.06
Ca/P	**2.17**	**2.01**	**1.95**	**1.90**	**45.33***	**2.05**	**2.02**	**1.81**

Results are the means ± SD (n = 3).
* Significantly different from the values detected in *allec* reference ($p < 0.05$) using ANOVA.

FIG. 5. *Minerals content (mg/g of ash) of* dolia, allec *reference and fresh anchovy samples.*

	D1	D2	D3	D4	D5	D6	*allec* reference
L*	46.8 ± 0.2*	49.1 ± 0.8*	54.6 ± 0.1*	42.4 ± 1.7	37.2 ± 0.4*	49.75 ± 1.3*	41.7± 0.2
a*	12.2 ± 0.2*	10.6 ± 0.2*	13.1 ± 0.2*	13.8 ± 0.8*	9.7 ± 0.9*	12.5 ± 1.1*	6.4 ± 0.7
b*	34.5 ± 3.3*	29.7 ± 0.8	36.4 ± 0.5*	35.8 ± 2.8*	18.3 ± 1.7*	34.1 ± 0.6*	29.9 ± 1.3

Results are the means ± SD (n = 3).
* Significantly different from the values detected in *allec* reference ($p < 0.05$) using ANOVA.

FIG. 6. *CIELAB colour parameters of* dolia *and* allec *reference samples.*

sample, with a Ca/P ratio of 45, indicate that the Ca present in this *dolia* is not only the result of fish bone, and it was added by either the Romans or by volcanic ash contamination. Some authors reported that volcanic ash from Vesuvius had a mineral composition of Ca (7-21%), Fe_2O_3 (4-9%), Na_2O (1-4%), K_2O (3-9%) (Belkin *et al.*, 1993; Horwell *et al.*, 2010). Therefore, attending to the relatively high content of Ca, K and Fe in D5, it is probable that this *dolia* was contaminated by volcanic ash. Nevertheless, this contamination could not explain the high Ca content in D5. In Roman times, lime (CaO) was used as an acidity neutralizing agent in *garum –liquamen–* and as an antiseptic agent used for the cleaning and sanitizing of amphora. This would explain the presence of an amphora full of lime found near the production site in the *Garum* Shop (Bernal *et al.*, 2014).

All the remains show Fe content –3.8-7.42 mg/g– higher than the fresh anchovy and *allec* reference –0.11 and 0.09 mg/g, respectively–, although previous researchers did not detect this mineral in D6 (Smriga *et al.*, 2010). As previously observed, the iron deposition could proceed from volcanic ashes that could have contaminated the *dolia* during the post-excavation phase. However, this is not probable due to the ratio of Ca/P and low K content found in the remains that were different to mineral composition of volcanic ashes, with the exception of D5 and D1. Therefore, the human addition of Fe from different sources during the *garum* preparation seems a plausible hypothesis. The use of ferric ore had been reported by Athanassopoulos (1925) that referred to the use of dust rich in ferric minerals (Fe_2O_3) as food coloring for salt-cured sardines. Other authors reported the use of Fe_2O_3 to dye the fish sauces as treatment for imitating the famous *garum haimation* or blood-*garum* (Van Neer and Parker, 2008; Bernal and Domínguez, 2011). Therefore the use of additives in Roman food could be a way to improve the appearance, imitate other foods or revalue the product. However, the Fe could also proceed from other organic sources like fish blood or viscera.

The hypothesis of Fe addition is supported by the color differences found between the archaeological remains and the *allec* reference (Fig. 6). CIELAB parameters indicate that all the remains, except D5, exhibit higher a* and b* values than the *allec* reference. They show a reddish-orange color, while the *allec* reference has a grayish color. Likewise, all *dolia* show higher luminosity –L*– than the *allec* reference, with the exception of D5.

4. Principal components Analysis (PCA)

PCA was applied to the composition variables –moisture, fat, nitrogen, ash, ∑SFA content, ∑MUFA content, ∑PUFAs content, Ca, P, K, Na, Fe, Mg, Cu and Zn– of archaeological remains to determine the statistical significance of differences between the factors. Three factors were extracted that accounted for 87.8% of the total variance within the data (Fig. 7). The percentage of variance explained is relatively high.

Factor 1 –34.3% of the total variance– was highly correlated with variables associated with the mineral contents –Ca, P, K, Fe and Zn–. It had a positive correlation with Ca, P and Zn, related to the mineral material from fish bone and had a negative correlation with K and Fe, related to other sources used in the elaboration process. Thus, the greater the Ca and P content of the remains, the smaller the Fe and K content, indicating more fish remains and

	Factor 1	Factor 2	Factor 3
Ash	-0.040645	-0.972392*	-0.066963
Total N	-0.082512	0.973717*	-0.036730
Fat	0.010823	0.968381*	0.006038
SFA	0.326717	-0.118386	-0.906975*
MUFA	-0.327644	0.127310	0.897392*
PUFA	-0.271094	0.056833	0.790983*
P	0.920810*	0.159353	-0.279191
Ca	0.830024*	-0.210367	-0.242931
Na	0.469919	0.776410*	0.105601
Mg	0.249436	0.825557*	0.359693
K	-0.917116*	-0.014078	0.280126
Fe	-0.747021*	-0.347290	0.481430
Cu	0.656947	0.124060	0.479724
Zn	0.926984*	0.138997	-0.211592
Exp. Var	4.806058	4.374409	3.122087
Prp total	0.343290	0.312458	0.223006

Fig. 7. *Results of factor loadings (varimax raw) Principal Components (marked loadings are > 0.700000).*

	Factor 1	Factor 2	Factor 3
D1	-0.65	1.63	-0.09
D2	0.89	0.85	-0.15
D3	0.41	-0.31	-1.59
D4	0.92	-0.96	0.94
D5	-1.71	-0.81	0.59
D6	0.14	-0.39	-0.98

Fig. 8. *Factor scores (varimax raw) Principal Components (marked loadings are > 0.700000).*

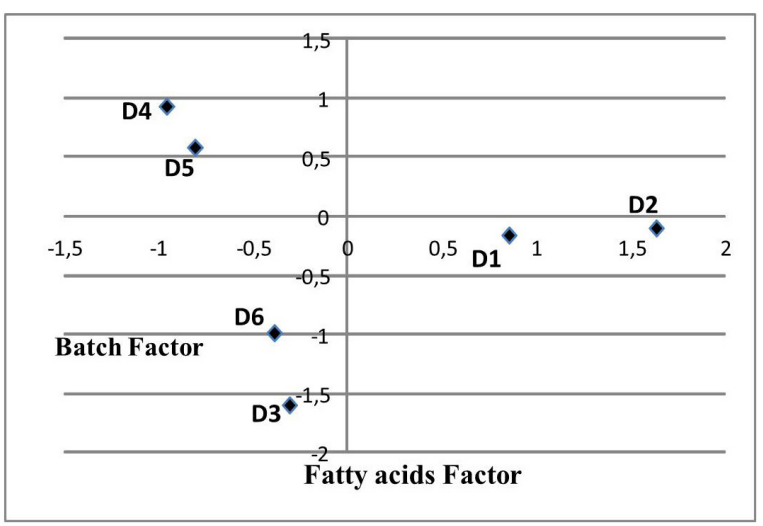

Fig. 9. *Distribution of* dolia *according to their factorial loads (F2 vs. F3) for batch and fatty acid factors.*

less contribution from other ingredients in the *dolia*. F1 is, therefore, a mineral factor that establishes the degree of deviation with respect to the mineral composition of the fish bone due to use of other ingredients. As seen in Fig. 8, only in D1 and especially in D5 with F1 negative, shows more deviation from the mineral composition of fish bone due to high presence of Fe in the *dolia*.

Factor 2 –31.2% of the total variance– was positively correlated with total N, fat, Na and Mg and negatively correlated with ash (Fig. 7). This factor indicates mainly the amount of organic matter present in the *dolia*, and it could establish some differences by batch in the raw materials used in *garum* production (Fig. 8). Therefore, this factor is referred to as the bath factor.

Factor 3 –22.3 % of the total variance– was positively correlated with unsaturated fatty acids –MUFA and PUFA– and negatively correlated with saturated fatty acids; hereinafter it is referred to as the fatty acid factor. This factor is also indicative of batch production. As seen in Fig. 8, D3 and D6 show high negative F2 values due mainly to the majority proportion of SFAs.

Representing the factorial loads of Factor 2 –F2– *vs.* Factor 3 –F3– of *dolia* (Fig. 9), it can be observed that these tend to be grouped according to the batch determined by the raw materials, ingredients and production stage, showing again three clusters that are significantly differentiated by the F2 and F3 axis values. Therefore, in the first cluster –F2 and F3 > 0– were included in the D1 and D2 *dolia* and were characterized by the preparation stage they were found in with the rest of *garum* and *allec* and ingredients that were distinct from those of the rest of the *dolia*. D3 and D6 were grouped into a second cluster –F2 and F3 < 0– characterized by a low content in organic matter, MUFA and PUFA, which means that *garum* was being prepared in these *dolia* but that it was a different batch. Finally, D4 and D5 –F2 < 0 and F3 > 0– were grouped

by their very low content in organic matter and in SFA, which is likely the result of the separation of *garum –liquamen–* and *allec*.

5. Conclusions

This study has served to implement a technological approach to determining the preparation methods and possible ingredients or additives used in ancient *garum* production. The chemical analysis performed on fish archaeological remains found in six vessels *–dolia–* at the '*Garum* Shop' of Pompeii (Italy), *allec* samples obtained following the reconstruction process, and fresh anchovy show that different *garum* batches were being manufactured at the moment of the Vesuvian eruption and that some of them could have been in different stages of the preparation process. D1, D2, D3 and D6 were containers being used for *garum* production; while D4 and D5 were being used to store the finished product: they had liquid *(liquamen)* and *allec* or both already separated.

The ratio of (∑SFAs + ∑MUFAs)/∑PUFAs shows similarities between *allec* reference and archaeological remains. The high C20:1, C24:1, DHA and EPA contents found in D1 and D2 could indicate the use of an additional fatty acids source other than that assumed by fish during the ancient *garum* manufacturing process in this batch.

The mineral analysis is useful to determine the possible use of Ca and Fe sources in the *garum* process as additives or their use for cleaning the surfaces and vessels.

PCA analysis contributed to the study through three factors: mineral –F1–, batch –F2– and fatty acids –F3–. Mineral factors established the degree of deviation with respect to the mineral composition of the fish bone due to presence of other ingredients or additives. Batch and fatty acid factors verified the *dolia* clusters according to the raw materials, ingredients and production stage.

Bibliography

AFNOR (1996): *Dosage des minéraux-méthodes par spectrométrie d'emission de flame*, NF-T-90-019. Paris: Association Française de Normalisation.

AOAC (2000; 17th ed.): *Official Methods of Analysis*. Arlington: AOAC *international*.

ATHANASSOPOULOS, G. (1925): "Use of 'terre rouge' in salting sardines; chemical composition of substances". In *Note sur la péche en Gréce.* Institute Oceanographic Bulletin, no. 454. Monaco: pp. 6-7.

BEDDOWS, C. G. (1998; 3rd ed.): "Fermented fish and fish products". In WOOD, B. J. B. (ed.): *Microbiology of Fermented Food*. New York: Blackie, pp. 1-39.

BEKKER-NIELSEN, T. (2005): *Ancient fishing and fish processing in the Black Sea region.* Black Sea studies, 2. Aarhus.

BELKIN, H. E.; KILBUM, C. R. J. and DE VIVO, B. (1993): "Chemistry of the lavas and tephra from recent (AD 131-1944) Vesuvius (Italy) volcanic activity", *U.S. Geol. Surv*, Open-File Rep, 44, pp. 393-399.

BERNAL, D. (2011): *Pescar con Arte. Fenicios y romanos en el origen de los aparejos andaluces*. Cádiz.

BERNAL, D. and COTTICA, D. (2010): *Pesca e Garum a Pompei ed Ercolano. Primi risultati di un progetto di ricerca interdisciplinare sullo sfruttamento delle risorse del mare in area vesubiana, Scienze Naturali e Archeologia. Il paesaggio antico. Interazione uomo-ambiente ed eventi catastrofici (Nápoles, octubre, 2010).* Napoli: Aracne.

BERNAL, D.; COTTICA, D.; BUSTAMANTE, M.; DÍAZ, J. J.; EXPÓSITO, J. A.; GARCÍA, E.; GÓMEZ, A.; LANDI, S.; LARA, M.; LORENZO, L.; MARLASCA, R.; RIQUELME, J. A.; RODRÍGUEZ, C. G.; SÁEZ, A. M.; VARGAS, J. and VERDUGO, J. (2013): *Pesca y garum en Pompeya y Herculano. Cuarta campaña arqueológica*. Informes y Trabajos, 9. Excavaciones en el exterior. Madrid: Ministerio de Cultura, pp. 322-339.

BERNAL, D.; COTTICA, D.; GARCÍA-VARGAS, E.; TONIOLO, L.; RODRÍGUEZ-SANTANA, C.; ACQUA, C.; MARLASCA, R.; SÁEZ, J. M.; VARGAS, J. M.; SCREMIN, F. and LANDI, S. (2014): "Un contexto excepcional en Pompeya: la pila de ánforas de la Bottega del Garum (I, 12, 8)", *Rei Cretariæ Romanæ Favtorvm*, Acta, 43, pp. 219-232.

BERNAL, D. and DOMÍNGUEZ, S. (2011): "Colorantes y pigmentos en las pesquerías hispanorromanas", *Cuadernos de Prehistoria y Arqueología de la UAM*, 37-38, pp. 671-685.

BERNAL, D. and SÁEZ, A. M. (2006): "*Infundibula gaditana*. Acerca de los vasos troncocónicos perforados para filtrar *garum* y otros usos industriales en la bahía de Cádiz", *Romula*, 5, pp. 167-218.

BOTTE, E. (2009): *Salaisons et sauces de poissons en Italie du sud et en Sicile durant l'Antiquité*. Collection du Centre Jean Bérard, 31. Naples.

Brown, L. D. and Heron, C. (2004): "Exploring links: preliminary investigations into marine resources and ceramics from Old Scatness, Shetland". In Housley, R. A and Coles, G. (eds.): *Atlantic Connections and Adaptations Economies, Environments and Subsistence in Lands Bordering the North Atlantic. Symposia of the Association for Environmental Archaeology*. Oxford: Oxbow Books, pp. 146-154.

Brown, T. A. and Brown, K. A. (1992): "Ancient dna and the archaeologist", *Antiquity*, 66, pp. 10-23.

Brown, T. A. and Brown, K. A. (2011): *Biomolecular Archaeology: an Introduction*. Chichester: Wiley-Blackwell.

Carannante, A. (2010): "L'ultimo garum di Pompei. Analisi archeozoologiche sui resti di pesce dalla cosiddetta Officina del garum", *Automata*, iii-iv (1), pp. 43-53.

Cha, Y. J.; Lee, E. H. and Kim, H. Y. (1985): "Studies on the processing of low salt fermented sea foods. Changes in volatile compounds and fatty acid composition during the fermentation of anchovy prepared with low sodium contents", *Bulletin of the Korean Fisheries Society*, 18 (6), pp. 511-518.

cie (1978): *International Commission on Illumination, Recommendations on Uniform Color Spaces, Color Difference Equations, Psychometric Color Terms. Suppl. 2 to cie publication no. 15 (e-1.3.1) 1971/(tc-1.3)*. Paris: Bureau Central de la cie.

Crisa, E. and Sands, A. (1975): "Microflora of four fermented fish sauces", *Applied Microbiology*, 29, pp. 106-108.

Curtis, R. I. (1979): "The garum shop of Pompeii", *Chronache Pompeiane*, 5, pp. 5-23.

Curtis, R. I. (1983): "In defense of garum", *The Classical Journal*, 78, pp. 232-240.

Curtis, R. I. (1991): *Garum and Salsamenta. Production and commerce in Materia Medica. Studies in ancient medicine. Vol. 3*. Leiden: Brill.

Curtis, R. I. (2009): "Umami and the foods of classical antiquity", *American Journal of Clinical Nutrition*, 90 (s), pp. 712-718.

Dallongeville, S.; Garnier, N.; Bernal, D.; Bonifay, M.; Rolando, C. and Tokarski, C. (2011): "Dealing with the identification of protein species in ancient amphorae", *Analytical and Bioanalytical Chemistry*, 399, pp. 3053-3063.

Desse-Berset, N. and Desse, J. (2000): "Salsamenta, garum et autres préparations de poissons. Ce qu'en disent les os", *Mélanges de l'École Française de Rome. Antiquité*, 112 (1), pp. 73-97.

Dieffenbacher, A. and Pockilngton, W. D. (1992): *International Union of Pure and Applied Chemistry (iupac), Applied Chemistry Division. Commission on Oils, Fats and Derivatives. Standard methods for the analysis of oils, fats and derivatives.* 1st suppl. to the 7th edition. London: Blackwell Scientific Publications.

Étienne, R. and Mayet, F. (2002): *Salaisons et sauces de poisson hispaniques.* Paris: Diffusion Édit. De Boccard.

Evershed, R. (1993): "Biomolecular Archaeology and Lipids", *World Archaelogy*, 25 (1), pp. 74-93.

Evershed, R. (2008): "Organic residues in archaeology; the archaeological biomarker revolution", *Archaeometry*, 50 (6), pp. 895-924.

García-Vargas, E. and Bernal, D. (2009). "Roma y la producción de *garum* y *salsamenta* en la costa meridional de *Hispania*. Estado actual de la investigación". In Bernal, D. (ed.): *Arqueología de la pesca en el Estrecho de Gibraltar*. Cádiz: uca.

García-Vargas, E.; Bernal, D.; Palacios, V. M.; Roldán, A. M.; Rodríguez, A. and Sánchez, J. (2014): "*Confectio gari pompeiani*. Procedimiento experimental para la elaboración de salsas de pescado romanas", *spal*, 23, pp. 65-82.

Garnier, N.; Richardin, P.; Cheynier, V. and Regert, M. (2003): "Characterization of thermally assisted hydrolysis and methylation products of polyphenols from modern and archaeological vine derivatives using gas chromatography-mass spectrometry", *Analytica Chimica*, 493, pp. 137-157.

Garnier, N.; Rolando, C.; Munk-Hotjec, J. and Tokarski, C. (2009): "Analysis of archaeological triacylglycerols by high resolution nanoesi, ft-icr ms and irmpd ms/ms: Application to 5th century bc-4th century ad oil lamps from Olbia (Ukraine)", *International Journal of Mass Spectrometry*, 284, pp. 47-56.

Grainger, S. (2011): "Roman fish sauces: an experiment in archaeology". In *Cured, Smoked, and Fermented fish. Proceedings of the Oxford Symposium on Food*. London: Prospect Books, pp. 121-131.

Grimal, P. and Monod, T. (1952): "Sur la veritable nature du garum", *Revue des Études Anciennnes*, 54, pp. 26-38.

Gurioli, L.; Pareschi, M. T.; Zanella, E.; Lanza, R.; Deluca, E. and Bisson, M. (2005): "Interaction of pyroclastic density currents with human settlements: evidence from ancient Pompeii", *Geology*, 33, pp. 441-444.

Horwell, C. J.; Stannett, G. W.; Andronico, D.; Bertagnini, A.; Fubini, B.; Le Blond, J. S. and

Williansom, B. J. (2010): "A physico-chemical assessment of the health hazard of Mt Vesuvius volcanic ash", *Journal of Volcanology and Geothermal Research*, pp. 222-232.

Ikeda, K. (2002): "New seasonings", *Chemical Senses*, 27, pp. 847-849.

Loftsson, T.; Ilievska, B.; Asgrimsdottir, G. M.; Ormarsson, O. M. and Stefansson, E. (2016): "Fatty acids from marine lipids: Biological activity, formulation and stability", *Journal of Drug Delivery Science and Technology*, 34, pp. 710-715.

Luongo, G.; Perrotta, A. and Scarpati, C. (2003): "Impact of the AD 79 explosive eruption on Pompeii. I. Relations amongst the depositional mechanisms of the pyroclastic products, the framework of the buildings and the associated destructive events", *Journal of Volcanology and Geothermal Research*, 126, pp. 201-223.

Martínez-Valverde, I.; Periago, M. J.; Santaella, M. and Ros, G. (2000): "The content and nutritional significance of minerals on fish flesh in the presence and absence of bone", *Food Chemistry*, 71, pp. 503-509.

Mika, A.; Swiezewska, E. and Stepnowski, P. (2016): "Polar and neutral lipid composition and fatty acids profile in selected fish meals depending on raw material and grade of products", *Food Science and Technology*, 70, pp. 199-207.

Mizutani, T.; Kimizuka, A.; Ruddle, K. and Ishige, N. (1992): "Chemical components of fermented fish products", *Journal of Food Composition and Analysis*, 5, pp. 152-159.

Palacios, V. M.; García, E.; Bernal, D.; Roldán, A.; Rodríguez, A. and Sánchez, J. (2016): "Conservas antiguas y gastronomía contemporánea". In Bernal, D.; Expósito, A.; Medina, L. and Franqueira, J. (eds.): *Un estrecho de conservas*. Cádiz: UCA, pp. 76-93.

Passi, S.; Rothschild-Boros, M. C.; Fasella, P.; Nazzaro-Porro, M. and Whitehouse, D. (2015): "An application of high performance liquid chromatography to analysis of lipids in archaeological samples", *Journal of Lipid Research*, 22, pp. 778-784.

Pecci, A.; Cau, M. A. and Garnier, N. (2013a): "Identifying wine and oil production: analysis of residues from Roman and Late Antique plastered vats", *Journal of Archaeological Science*, 40, pp. 4491-4498.

Pecci, A.; Giorgi, G.; Salvini, L. and Cau, M. A. (2013b): "Identifying wine markers in ceramics and plasters using gas chromatography-mass spectrometry. Experimental and archaeological materials", *Journal of Archaeological Science*, 40, pp. 109-115.

Ponsich, M. and Tarradell, M. (1965): *Garum et industries antiques de salaison dans la Méditerranée occidentale*. Société d'Études Latines de Bruxelles, 3. Bruxelles, pp. 711-713.

Reese, D. S. (2002): *Fish: evidence of specimens, mosaics, wall painting, and Roman authors*. The Natural History of Pompeii, 12. Cambridge: CUP, pp. 274-291.

Regert, M. (2011): "Analytical strategies for discriminating archaeological fatty substances from animal origin", *Mass Spectrometry Reviews*, 30, pp. 177-220.

Regert, M.; Garnier, N.; Decavallas, O.; Cren-Olivé, C. and Rolando, C. (2003): "Structural characterization of lipid constituents from natural substances preserved in archaeological environments", *Measurement Science and Technology*, 14, pp. 1620-1630.

Ruddle, K. and Naomichi, I. (2010): "On the origins, diffusion and cultural context of fermented fish products in Southeast Asia". In *Globalization, Food and Social Identities in the Asia Pacific Region*. Tokyo: Sophia Univ. Institute of Comparative Culture, pp. 1-17.

Smriga, M.; Mizukoshi, T.; Iwahata, D.; Eto, S.; Miyano, H.; Kimura, T. and Curtis, R. I. (2010): "Amino acids and minerals in ancient remnants of fish sauce *(garum)* sampled in the 'Garum Shop' of Pompeii, Italy", *Journal of Food Composition and Analysis*, 23, pp. 442-446.

Toppe, J.; Albrektsen, S.; Hope, B. and Aksnes, A. (2007): "Chemical composition, mineral content and aminoacid profiles in bones from various fish species", *Comparative Biochemistry and Physiology*, 146 (B), pp. 395-401.

Van Neer, W. and Parker, St. (2008): "First archaeozoological evidence for haimation, the 'invisible'garum", *Journal of Archaeological Science*, 35, pp. 1821-1827.

Zlatanos, S. and Laskaridis, K. (2007): "Seasonal variation in the fatty acid composition of three Mediterranean fish sardine *(Sardina pilchardus)*, anchovy *(Engraulis encrasicholus)* and picarel *(Spicara smaris)*", *Food Chemistry*, 103, pp. 725-728.

