Identifying spathic calcite recipe in archaeological ceramics: possibilities and limitations

(Indentificando calcita espática em cerâmicas arqueológicas: possibilidades e limitações)

D. A. Santacreu
Department of Historical Sciences & Arts Theory, University of the Balearic Islands, Spain
Ctra. Valldemossa km 7.5 s/n. Facultat de Filosofia I Lletres, Edifici Ramon Llull, Campus UIB, 07122, Mallorca, Spain
d.albero@uib.es

Abstract

In this paper we approach the study of a ceramic recipe based on the addition of crushed spathic calcite as temper. We first briefly discuss the anthropological significance underlying the identification of technological paste recipes, as well as the spatial distribution of the spathic calcite recipe in the Mediterranean basin. Subsequently, we focus on the possibilities of identifying this recipe using several analytical methods, including binocular microscopy, image analysis, petrology, X-ray powder diffraction and X-ray fluorescence. Thus, the complementarity existing between different methods commonly applied in pottery analysis is considered in order to know the potential of these various techniques to identify spathic calcite recipe and classify the ceramic record to maximize the social archaeological interpretation of the data. The final objective is to know the feasibility and limitations that certain analytical procedures - such as the binocular microscopy- have for extrapolating the identification of this recipe to a larger series of ceramics from only certain features of the paste.
Keywords: pottery recipes, spathic calcite, X-ray powder diffraction, X-ray fluorescence, petrology, image analysis, binocular microscopy.

Resumo

Um procedimento cerâmico baseado na adição de calcita espática moída como um dos procedimentos da pasta tecnológica é apresentado, assim como a distribuição espacial da calcita na bacia do mediterrâneo. Na seqüência, focalizamos nas possibilidades de identificar este procedimento usando vários métodos analíticos, incluindo microscopia binocular, análise de imagem, petrologia, difração de raios X e fluorescência de raios X. Assim, a complementaridade que existe entre os diferentes métodos comumente aplicado em análise de cerâmicas é considerada para conhecer o potencial dessas várias técnicas na identificação de procedimentos de calcita espática e classificar os dados cerâmicos para maximizar a interpretação arqueológica social dos dados. O objetivo final é conhecer a possibilidade e as limitações de certos procedimentos analíticos - tal como a microscopia binocular - tem para extrapolar a identificação destes procedimentos a uma ampla série de cerâmicas desde somente certos aspectos da pasta.
***Palavras-chave**: procedimentos cerâmicos, calcita espática, difração de raios X, fluorescência de raios X, análise de imagem, microscopia binocular.*

INTRODUCTION

When potters produce a vessel, they make different technological choices that are embodied in recipes, which involve using specific raw materials in relatively constant proportions [1, 2]. Using recipes in pottery production, for instance, is well documented ethnographically among potters from the Bantuethno-linguistic groups in South Africa [3], St. Nicholas in the Philippines [4] or Ancash in Peru [5]. As observed ethnographically in India [6], recipes used to produce ceramics go beyond environmental, technical, functional as well as economic factors and also have social and cultural value. Thus, recipes are strongly related to a set of social norms that regulate the manufacturing process and are consequence of the knowledge acquired by individuals as a result of technological and social behavior. The existence of common phenomenology is also evident in the use of recipes, which can remain stable over long periods of time, as they are closely related to certain social praxis, experiences and ways of doing that individuals tend to internalize in certain *habitus* [7]. In this sense, using specific materials and procedures can work - whether consciously or unconsciously - as an agent capable of strengthening the bonds of identity and social cohesion among members of the same community [2, 8, 9] in a process of materializing the cultural tradition.

Furthermore, recipes are the basis for human action, this assumption involves an active vision of technology in the society where individuals, in their technological choices, can act and express themselves socially through praxis and techniques associated with the use of certain materials. This agency framework explains the existence of variations in time and space in the composition of ceramic recipes, which affect, for instance, the kind or quantity of materials used. The continuation or rejection of a recipe can indicate changes in social structure, learning systems and knowledge transfer, as well as in people's rational schemes [7, 10, 11]. Using the concept of recipe in the study of pottery therefore allows anthropologists and archaeologists to move beyond the mere description and classification of ceramic assemblages and use a concept closely linked to the technological choices that potters apply in their daily life [12].

Approaching spathic calcite recipe

With the previous theoretical background in mind, in this paper we focus on the methodological development associated with a specific ceramic recipe that has a wide spatial and chronological distribution, namely, the one that uses crushed spathic calcite to temper the paste. In this way, spathic calcite is a commonly used temper in pottery in many societies and has a wide range of physical connotations that affect all the stages of the production process and the final product obtained, as well as the ceramic uses [8, 13, 14].

It can be considered - just as occurs with the use of oil in the Mediterranean cuisine - that the addition of spathic calcite crystals was a key element in most of the recipes used to produce ceramics in the Mediterranean basin along prehistory, among many other areas. In this way, the use of crushed calcite temper in surrounding regions could indicate the existence of contacts, social interactions and cultural transmission that would have promoted the establishment of a cultural *koine* throughout this large territory. Thus, the use of this temper is well-known in the Mediterranean area since the Neolithic (5000-3000 BC) and Chalcolithic (3000-1750 BC) periods in places like Greece [15], the Balkans [16], Italy [17, 18], southern France [19-22], and the Iberian Peninsula [23, 24]. Moreover, its use is also documented in the English Bell-Beaker period [25, 26]. During the Bronze and Iron Ages, spathic calcite recipe is widely documented in the Iberian Peninsula [24, 28-30], Balearic Islands [9, 31-33], Italy [34, 35], Belgium, and the area stretching from Burgundy to the Mediterranean [36]. Its use is also recognized in other parts of Central and Eastern Mediterranean, the Adriatic Sea, the Balkans [16] and Tell Mishrifeh/Qatna in Syria [37]. Finally, calcite was a common temper in Huntcliff ware from northern England during the Late Roman Empire [25].

As seen, spathic calcite ceramic recipe is well established in the Western Mediterranean area as technical feature as early as the Middle Neolithic, resulting in a common way to process different raw materials and prepare the paste, which seems to have occurred simultaneously in different places. Nevertheless, within the vast number of places and regions in which this mineral was available, the addition of crushed calcite is documented only in specific geographic areas. Some authors suggest [24] that these areas appear clearly opposed to others where such materials were also abundant but were not used. This dichotomy in the technological choices may evidence the coexistence of different technological traditions. Thus, it can be assumed that at least some potters might have known of the existence of other ceramic technologies and recipes related to other ways of making pottery. This fact could favor the establishment of certain identity elements: those not only structured on social and cultural similarities but also on differences with other potters' communities [38, 39].

Identification of spathic calcite recipe

On the whole, approaching the role of ceramic recipes in ancient societies requires establishing first the chronological and spatial distribution that recipes had in order to subsequently and properly study social and identity phenomena. Thus, it is within this theoretical and archaeological context where an intense methodological work focused on the identification of spathic calcite recipe must be achieved with the aim to advance in long-term research. In this way, the composition of ceramic bodies reflects the recipes used in manufacturing processes and the application of archaeometric techniques to pottery study provides information about the raw materials selected and the way pastes were prepared. Hence, taxonomic classification of the components and textures of ceramic bodies becomes an important process in studying the pottery technological choices and recipes. However, a proper classification of ceramic bodies - i.e. capable of developing its full potential and usefulness in the study of the spatial and temporal distribution of ceramics recipes -implies the study of large series of samples assigned to different archaeological contexts.

An important aspect in the classification of ceramic assemblages and the study of the technology of archaeological pottery relates to the characterization of the coarse fraction of the paste. The study of this fraction is usually quite accessible for researchers and archaeologists using optical methods. In this regard, petrographic analysis - focused on sample mineralogy and texture - is capital in establishing a proper classification of the ceramic coarse fraction. Unfortunately, we must be aware that the available funding, human and material resources usually restrict the number of petrographic analyses that can be performed, thus limiting the analytical possibilities in any archaeometric research. In contrast to petrographic analysis, the observation of pottery samples through binocular microscope is much more accessible. Nevertheless, the mineral identification using this technique is often ineffective and may have a high error rate. Furthermore, such approaches might have some problems in developing systematic descriptions of pottery pastes and normally generate only qualitative data mainly referring to the coarsest fraction of the ceramic body [28, 40, 41].

Despite these limitations, some scholars [27, 42-44] suggest that by studying the textural and optical characteristics of the ceramic pastes through binocular microscope and testing their correspondence with petrographic, mineralogical and chemical features, some results can be extrapolated to a larger number of pottery vessels. Thus, this procedure may allow an initial classification of ceramic assemblages without requiring the application of more specialized analytical techniques. Towards this direction, this paper aims to evaluate the possibilities and limitations that several traditional methods -including binocular microscopy - have in identifying and classifying ceramic bodies associated with the spathic calcite recipe. Hence, our goal is to recognize the potential that techniques such as binocular microscopy, petrographic analysis, X-ray powder diffraction (XRPD) and X-ray fluorescence (XRF) have in the identification of certain ancient potters'praxis and the classification of the ceramic record in order to maximize social archaeological interpretation of data derived from archaeological ceramics.

MATERIALS AND METHODS

Almost a hundred ceramics have been selected from different prehistoric periods and archaeological sites in Mallorca (Balearic Islands, Spain) (Table I).

Samples from this region were selected because, as stated before, this recipe is well documented in diverse eras and has a wide distribution within the island. We considered 92 hand-made and low-fired ceramics recovered from the excavations carried out in the archaeological sites of Closos de Can Gaià (Felanitx), Puig de Sa Morisca and Turó de les Abelles (Calvià). These settlements cover an extended time range, from the Early Bronze to the Late Iron Ages. Sample selection considered - macroscopically and under binocular microscopy -pastes with different textures and appearance features.

We selected 10 pottery samples from the Navetiforme I (boat-shaped structure) of the Bronze Age site of Closos de Can Gaià [45] dated in the Early Bronze Age (cal. 1760-1610 BC, KIA-11221), the Middle Bronze Age (cal. 1420-1250 BC, KIA-11233) and the Late Bronze Age (cal. 1040-840 BC, KIA-11232). We selected 46 samples from Tower I and Tower III of the prehistoric fortified site of Puig de Sa Morisca [46]. Samples from the Late Bronze Age (cal. 1120-920 BC, KIA- 33825), the Early Iron Age (cal. 750-400 BC, KIA- 33609) and primarily the Late Iron Age (cal. 410-350 BC, KIA- 33826) were selected from this location. In addition, 35 pottery samples from the Turó de les Abelles site were selected. The wheel-shaped ceramic materials found at this latter location suggest that was occupied in the Late Iron Age, between *c.*250-75 BC [47].

The methodology used to assess the presence of crushed spathic calcite forming part of the ceramic bodies has combined several analytical methods: XRF, XRPD, petrology, image analysis and binocular microscopy. These methods provide different information about pastes and their application seek to relate multiple data associated with diverse aspects of the ceramic pastes, from their basic features to their mineralogical, chemical, petrographic and textural composition.

Microscopic optical analysis was performed on 91 ceramics observed under standardized light conditions and in fresh fractures, using a Heerbrugg Wild binocular microscope with a lens range between 6 and 50x and a VolpiIntralux 5000 halogen lamp. In addition to the optical exam, a quantitative textural analysis through image analysis was conducted by combining the binocular microscope, a Leica DFC-320 Digital Camera and the image analysis software Leica Qwin. The Digital Image Analysis was made by capturing fresh cross-sections of pottery samples in digitally calibrated microphotographs with a resolution of 2000 dpi. Following the Delesse's principle - which states that the ratio of the area occupied by a component relative to the entire profile area is a consistent estimate of the volume fraction of the component in the object - we calculated the percentage (in volume) of all the non-plastic particles in the pastes over 50 μm in length using the formula specified by García del Amo [48]. Furthermore, the maximum length parameter was used to estimate the apparent particle size. The apparent particle sizes were then transformed infrequencies by classes which were considered for analysis. The numerical ranges for each class interval were based on a logarithmic scale of 50 to 4.000 μm (see [9] for a detailed description of the method).

Though binocular microscopy allows a basic approach to paste features, texture and chromatic transitions in the cross-sections, petrographic examination of the samples in thin sections is the best way to identify minerals that are part of the coarse paste fraction. Thus, this method allows determining the habit of the minerals, their appearance and morphology, something especially important when studying spathic calcite recipe, as it allows establishing whether the features of the crystals correspond to a natural origin or were intentionally added by potters. We have used thin sections in a selection of 34 of the pottery vessels -which showed different appearance features and textures -for optically-based mineralogical and textural studies. The amount of each compound in thin section was established using comparative charts [49]. The optical examination of samples was performed using Leica DM-RX and Olympus BX-60 petrographic microscopes that incorporate color filters and micrometers. The lenses used varied from 16x to 400x. Photomicrographs of the samples were taken with a Leica DC500 digital camera.

The addition of crushed spathic calcite in the samples must necessarily have a correlation in the concentrations of various minerals present in the paste. To verify the correspondence between optical observations, petrographic analysis and mineralogical composition of the pastes, 91 ceramics have been analyzed using X-ray powder diffraction (XRPD). Measurements were performed in a Siemens D5000 diffractometer using Cu Kα radiation (λ= 1.5405Å) and a monochromator gonium in the diffracted beam (at 45 kV and 40 mA). Spectra were taken from 3 to 70° at a rate of 3s per step. X-powder and Eva software were used to evaluate the crystalline phases according to the intensity and spaced tables from the data bank drawn from the Joint

Table I - Sample, period (EBA = Early Bronze Age, MBA = Middle Bronze Age, LBA = Late Bronze Age, EIA = Early Iron Age, LIA = Late Iron Age), percentage of mineral inclusions, CaO, calcite and spathic calcite recorded with the different methods for each sample analyzed.

[Tabela I - Amostra, período (EBA = era primeira do bronze, MBA = era média do bronze, LBA = era do bronze antiga, EIA = era primeira do ferro, LIA = era antiga do ferro) - porcentagem de inclusões minerais, CaO, calcita e calcita espática com dados de diferentes métodos para cada amostra analisada.]

Sample	% Mineral inclusions	*wt.*% CaO	Textural Group	Chronology	% Calcite (RIR method)	Spathic calcite
CG-1233	12.3	35.12	B	LBA	70.3	25
CG-149	4.1	15.76	C	EBA	33.4	10
CG-154	2.6	20.02	C	EBA	63.3	-
CG-1594	17.5	46.55	A	LBA	64.1	50
CG-160	5.1	25.47	C	EBA	35.2	-
CG-204	14.0	36.85	A	MBA	71.2	-
CG-219	17.2	43.73	A	MBA	86.2	-
CG-2664	19.1	49.44	A	LBA	86.2	40
CG-2755	3.9	3.13	C	LBA	3.0	0
CG-2795	8.3	13.46	C	EBA	6.8	-
SM-1125	17.0	38.46	A	LBA	90.9	30
SM-138	5.6	33.00	C	LIA	73.8	20
SM-140	16.8	29.85	A	LIA	23.5	0
SM-180	16.2	34.18	A	LIA	55.7	0
SM-181	12.2	27.98	B	LIA	35.8	0
SM-198	4.9	43.25	C	LIA	82.5	-
SM-238	6.9	34.79	C	LIA	84.9	-
SM-246	4.2	20.79	C	LIA	42.2	0
SM-280	5.1	46.03	C	LIA	74.9	-
SM-284	15.7	39.71	A	LIA	78.7	-
SM-293	4.4	17.52	-	LIA	39.5	-
SM-325	10.4	49.24	B	LIA	86.2	30
SM-36	3.1	3.62	C	LIA	3.8	-
SM-362	2.5	27.20	C	LIA	60.0	15
SM-386	21.0	44.09	A	LIA	95.1	-
SM-394	2.9	26.41	C	LIA	40.8	-
SM-416	6.7	41.15	C	LIA	56.2	-
SM-426	12.3	39.25	B	LIA	76.6	-
SM-468	28.0	32.09	E	LBA	48.0	45
SM-470	8.6	44.60	C	LBA	86.6	-
SM-471	2.9	30.55	D	LBA	72.9	-
SM-472	18.3	34.05	A	LBA	68.2	30
SM-473	22.4	38.43	A	LBA	64.3	35
SM-475	12.1	36.68	B	LBA	85.1	-
SM-484	17.2	35.41	A	LBA	58.0	30
SM-495	14.5	37.54	A	LBA	49.7	-
SM-625	17.5	11.34	A	LBA	19.3	-
SM-627	19.2	48.89	A	LBA	94.9	-
SM-634	14.9	32.71	A	LBA	82.9	-
SM-642	2.0	39.58	C	LIA	72.2	30
SM-659	3.9	25.47	C	LIA	39.6	15
SM-660	16.8	48.24	A	LIA	65.1	30
SM-661	22.3	41.59	A	LIA	62.7	35
SM-663	3.4	38.98	C	LIA	83.2	-
SM-665	14.3	38.12	A	LIA	79.5	30
SM-683	8.3	34.04	-	EIA	72.1	-
SM-697	11.4	34.58	B	LIA	81.1	-
SM-705	7.6	25.20	C	LIA	49.7	-
SM-708	8.6	41.24	C	LIA	59.7	-
SM-796	21.8	42.11	A	LIA	93.2	35
SM-823	9.0	42.84	C	LIA	66.4	-

Continuação da Tab. I
[Continuation of Tab. I]

Sample	% Mineral inclusions	*wt.*% CaO	Textural Group	Chronology	% Calcite (RIR method)	Spathic calcite
SM-834	9.3	53.65	C	LIA	93.3	-
SM-837	13.8	40.75	A	LIA	90.5	-
SM-970	21.4	43.47	A	LIA	52.8	35
SM-971	18.6	40.77	A	LIA	58.4	35
SM-1351	15.5	38.58	A	EIA	89.3	30
TSB-1.294	0.7	5.23	C	LIA	17.3	2
TSB-1.295	6.5	24.11	C	LIA	52.4	-
TSB-1.296	11.6	18.38	B	LIA	47.5	-
TSB-1.297	2.3	19.99	C	LIA	63.4	-
TSB-1.299	4.5	21.00	C	LIA	41.2	-
TSB-1.300	1.0	13.83	C	LIA	23.8	-
TSB-1.303	5.3	25.53	C	LIA	72.3	-
TSB-17.23	4.5	3.03	C	LIA	2.0	0
TSB-17.25	4.6	10.35	C	LIA	51.6	-
TSB-2.40	3.0	14.22	C	LIA	27.6	-
TSB-2.41	1.5	18.17	C	LIA	42.0	-
TSB-2.42	2.0	11.82	C	LIA	54.7	-
TSB-5.19	2.2	15.61	C	LIA	28.0	-
TSB-5.20	7.9	11.49	C	LIA	54.2	-
TSB-6.65	0.7	25.37	C	LIA	46.2	5
TSB-6.66	7.8	20.99	C	LIA	72.9	-
TSB-6.72	2.9	9.40	C	LIA	42.0	0
TSB-6.73	0.5	4.90	C	LIA	6.8	-
TSB-6.79	16.7	26.58	A	LIA	89.7	-
TSB-6.80	2.7	8.23	C	LIA	27.1	5
TSB-6.81	1.3	9.95	C	LIA	33.1	-
TSB-6.82	8.7	9.17	B	LIA	56.5	15
TSB-6.87	3.6	8.63	C	LIA	72.6	-
TSB-7.10	5.1	15.87	C	LIA	26.9	-
TSB-7.159	1.1	2.17	C	LIA	1.0	-
TSB-7.32	4.6	17.36	C	LIA	49.3	-
TSB-8.32	4.1	15.06	C	LIA	31.0	-
TSB-8.34	8.9	9.77	C	LIA	89.5	-
TSB-8.65	2.5	20.68	C	LIA	48.9	-
TSB-9.14	2.2	9.33	C	LIA	51.4	10
TSB-9.80	10.6	18.89	B	LIA	53.8	15
TSB-9.81	4.6	14.88	-	LIA	83.5	-
TSB-9.84	4.9	20.64	C	LIA	83.1	-
TSB-9.90	2.7	31.54	C	LIA	69.8	-
TSB-9.91	1.7	34.79	C	LIA	58.7	-

Committee of Powder Diffraction Standards. Finally, a semi-quantification of phases was achieved using the normalized Reference Intensity Ratios method [50, 51].

Finally, since calcite is CaCO$_3$ the intentional addition of this mineral to the paste should provide higher concentrations of CaO in the ceramic bodies. Exploring the chemical composition and CaO percentage estimations of all pottery samples was achieved using X-ray fluorescence (XRF). Samples were prepared by pulverization and furnace-drying at 80 ºC for 4 h, pressed pills were made using 1.5 g of the powder. The analysis was performed in an X-ray spectrometer Philips MagiX Pro-PW2400, while qualitative analysis was carried out using SuperQ software. The measurement of the major, minor and trace elements and oxides were conducted in a helium atmosphere supported in a polyester film (Mylar®). Quantification of the samples was normalized to 100% and achieved using IQ+ software.

RESULTS

Implementing the various methods described above has led to different kinds of evidence for the addition of crushed spathic calcite to the paste. On the one hand, direct evidence was stated using optical methods. On the other hand, we refer to indirect evidence when the addition of this mineral can be estimated from an abundance criterion using non-optical methods such as XRF or XRPD. Applying a variety of methods has also revealed certain limitations in macroscopic examination, binocular microscope observations and indirect methods (XRPD and XRF) when discriminating between this and other technological solutions made by potters.

Direct evidence: petrology and binocular microscopy

Petrographic study has allowed characterizing a series of morphological features of the crushed spathic calcite that can easily be observed, albeit partially, using a binocular microscope (Figs. 1a and 1b).

The calcite temper detected petrographically is characterized by a crystalline habit that - when not thermally altered - presents translucent spathic and monocrystalline crystals with prismatic and rhombohedra morphology, normally up to 1500 μm in size. These crystals are euhedral and have well-defined sharp angles and planes, which is rare in a natural state [52, 53]. Based on the crystalline character, the high amount of mineral present and the angular and rhombohedra morphology of the particles, we can infer that potters intentionally added crushed spathic calcite to the paste in many samples in varying proportions. Estimations of the frequency undertaken by means comparative charts showed that this mineral was added up to 40% in some ceramics, while others have up to 10%; the most common range in pottery samples is between 20-30%.

Under the binocular microscope, samples with a large amount of spathic calcite present abundant white or gray translucent crystals that often have an angular, rhombohedral and prismatic morphology. These must undoubtedly be associated with the spathic calcite crystals identified in thin section under the petrographic microscope. Samples that have this mineral in large quantities and sizes can even be identified macroscopically, as many gray or white crystals can be observed in the surface of the pottery, but especially in its fresh cross-section. Thus, the correspondence of the calcite temper features in both techniques provides a viable approach to suggest the presence of this added mineral without requiring thin sections of all pottery samples recovered in the archaeological excavations. Some scholars [33, 42] already observed this fact in their analysis of prehistoric pottery from Mallorca and have also proposed it as a valid procedure. After all, as Orton, Tyers & Vince [54] stated, by comparing the thin section features with a visual examination of the samples, we can understand how the petrographic traits correspond to certain observable macroscopic and microscopic characteristics. This comparison then increases the value of both analytical methods.

Statistical analysis of the textural data obtained from the ceramic pastes demonstrates a significant positive correlation coefficient between the quantity of crushed spathic calcite petrographically documented - established by comparative charts - and mineral inclusions determined by image analysis and binocular microscopy (*r Pearson* = 0.714, *n* = 34, significance 0.99). In addition, the determination of quantitative parameters for the texture of the vessels by means of image analysis permitted the establishment of three broad textural groups through cluster analysis according to the percentage of non-plastic mineral phase (in volume) and the apparent particle size present in the samples (see [9] for a detailed description of the analysis). The first textural group

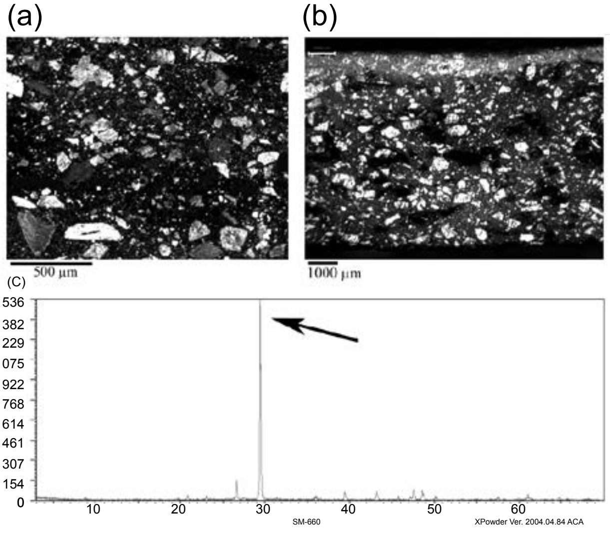

Figure 1: Pottery sample SM-660. A) Microphotograph taken by means of binocular microscope showing a high percentage (30%) of white and grey coarse rhombic particles. B) Microphotograph taken by means of petrographic microscope showing high amount of spathic calcite crystals. C) XRPD diffractogram showing a high intense peak of calcite at 3.03 Å.

[Figura 1: Amostra cerâmica SM-660. A) Fotomicrografia obtida em microscópio binocular mostrando alta porcentagem (30%) de partículas romboédricas cinzas grossas. B) Fotomicrografia obtida em microscópio petrográfico mostrando alto teor de cristais de calcita spática. C) Difratograma de raios X mostrando o pico de alta intensidade da calcita em 3 Å.]

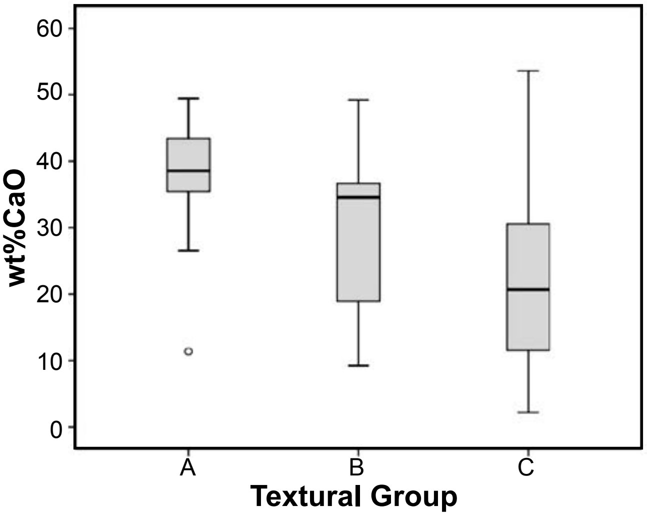

Figure 2: Boxplot showing differences in the % of CaO between the three main textural groups established by means of image analysis and binocular microscopy.

[Figura 2: Gráfico mostrando as diferenças na porcentagem de CaO entre os três principais grupos texturais estabelecidos por análise de imagem e microscopia binocular.]

(TG-A, Fig. 1b) is characterized by the highest amount of mineral phase (14-22%) and coarsest grain size (>800 μm). The second textural group (TG-B, Fig. 4d) is characterized by moderate amounts of mineral phase (9-13%) containing coarse or medium-sized particles (>300 μm). Finally, the last textural group (TG-C, Fig. 4f) is characterized by medium (5-9%) and especially low (<3%) percentages of mineral phase. In this case, coarse-textured pottery vessels are absent and the particle size is finer, mainly up to 800 μm and under 300 μm in most samples. We appreciated that it is easy to establish -using a binocular microscope and following an extensive petrographic examination - the presence of spathic calcite in most samples that have textures of type TG-A and TG-B. It is more difficult to identify such crystals in pastes of the TG-C group where, if present, they are in a much less significant number.

Indirect evidence: XRF and XRPD

Pastes with larger amounts of spathic calcite (>20%) in thin sections have also intense calcite peaks at 3.03 Å in the X-ray powder diffractograms (Fig. 1c), as well as high percentages of this mineral (>40%) in the semi-quantification of mineral phases performed. As expected, pottery samples with these characteristics have shown, in turn, a high CaO content (>20%) in the chemical analysis performed using XRF. This correspondence is expressed clearly in a significant positive correlation between the percentage of CaO determined by XRF and the percentage of calcite determined by XRPD (*r Pearson* = 0.715, *n* = 91, significance 0.99).The same phenomena occurs if petrographic estimations are related to CaO concentration (*r Pearson* = 0.785, *n* = 34, significance 0.99) and the calcite percentage determined by XRPD (*r Pearson* = 0.715, *n* = 34, significance 0.99).

This correlation decreases significantly if the CaO percentage is compared to the percentage of mineral inclusions determined by image analysis (*r Pearson* = 0.605, *n* = 91, significance 0.99). However, it must be said that both variables are difficult to compare, since two reasons may condition the gap existing between them. Firstly, part of the CaO and calcite percentages recorded in the samples must be also related to the use of calcareous clays for making most pottery vessels [9, 13]. Secondly, it should be considered the presence of post deposition almicritic calcite -associated with a completely allochthonous origin - that was introduced into the ceramic bodies during their burial. This kind of postdepositional phase has been observed in both, the surface and the cross-section of most of the samples analyzed here [55].

Despite these limitations, chemical and mineralogical analysis provides us with an indirect procedure that indicates the presence and amount of calcite temper added to the paste. Thus, the presence of large quantities of crushed spathic calcite in the pastes causes a significant increase in the amount of calcium oxide and calcite present in the chemical and mineralogical analysis, respectively. The presence of this temper has been well documented by petrographic examination, but also through the combination of the XRPD and/or XRF data with binocular microscope observations of the appearance of the ceramic bodies and the application of an abundance principle. The addition of this mineral is clear, since such CaO and calcite levels are not common in the natural state of the clays found in the Balearic Islands [32, 56].

Under the binocular microscope, pastes with high CaO and calcite concentrations have showed abundant translucent white or gray crystals that often exhibit rhombic and prismatic morphology. These must undoubtedly be associated with the spathic calcite crystals identified under petrographic microscope. In addition, there is some correspondence between the chemical and mineralogical data with the textural groups established by means of image analysis according to the grain-size and percentage of inclusions present in the samples. In this sense, it can be seen how the textural groups characterized by a mineral inclusion percentage over 10% (in volume), with the particle size over> 800 μm being the highest area percentage within the ceramic matrix (i.e. TG-A and to a lesser extent TG-B) are the ones which show the highest average amount of % CaO and calcite (Table II, Fig. 2).

In contrast, the Textural Group C - which is characterized by a finer texture - has significantly lower concentrations of % CaO and calcite. Nevertheless, while there is a general trend towards the higher the number of coarse inclusions the greater the percentage of % CaO and calcite, it is also certain - as seen in the high standard deviations recorded - that the values obtained are highly variable in all the textural groups (Table II).These high standard deviations should be related to the two phenomena discussed above: 1) variations in the more or less calcareous nature of the clays selected to produce pottery and 2) the presence of post depositional calcite which was incorporated into the vessels during their burial. Both phenomena may increase the amount of micritic calcite in the paste - and therefore the % of CaO and calcite -, thus obscuring the identification of the spathic calcite recipe by using only non-optical methods such as XRPD and XRF.

In spite of the difficulties highlighted the data obtained through the combination of these two methods with binocular microscope is strong enough to develop a proper ceramic classification based on the occurrence of spathic calcite recipe. Thus, the presence under binocular microscope of crystals with the characteristics described above in conjunction with high CaO or calcite concentrations detected by simple and inexpensive methods - including hydrochloric acid or calcimetry [31] - can be used to sort the ceramic assemblages and face relevant archaeological concerns. Moreover, other methods-such as X-ray diffraction and X-ray fluorescence - which are often applied in the analysis of archaeological ceramics for other purposes (e.g. studying the provenance of the raw materials or the firing strategies) can be also applied to reach this objective. In short, this procedure - which combines petrographic, mineralogical and chemical analysis with binocular microscope observations

Table II - Descriptive statistical analysis based on measures of central tendency regarding the % CaO and calcite present in the fabrics according to the different Textural Groups established by means of image analysis.
[Tabela II - Análise estatística descritiva baseada em medidas de tendência central em função da porcentagem de CaO e calcita presente nos tecidos de acordo com os diferentes Grupos Texturais estabelecidos por meio de análise de imagem.]

		Mean	Maximum	Minimum	Standard deviation
Textural Group A	%CaO	38.4	49.4	11.3	8
	%Calcite	70.8	95.1	19.3	20.7
Textural Group B	%CaO	29.9	49.2	9.1	12.4
	%Calcite	65.8	86.2	35.8	18.1
Textural Group C	%CaO	21.7	53.6	2.1	13
	%Calcite	49.5	93.3	1	25

- has allowed us to relatively safe extrapolate the addition of high amounts of spathic calcite crystals in a significant number of pottery vessels without requiring thin sections (e.g. CLG-204, CLG-219, SM-198, SM-238, SM-280, SM-284, SM-386, SM-426, SM-470, SM-471, SM-475, SM-495, SM-627, SM-634, SM-663, SM-683, SM-697, SM-705, SM-708, SM-834, SM-837, TSB-1/303, TSB-6/66, TSB-6/79, TSB-9/80, TSB-8/34, TSB-7/32, TSB-9/84).

Limitations

The macroscopic observations and the approaches made by means of binocular microscope may have some limitations, especially if other techniques -including the specified petrographic, mineralogical and chemical methods - are not applied in the ceramic analysis. As Daszkiewiczet al. [43, 30, 31] pointed out *Multiple laboratory analyses have revealed that the white grains macroscopically interpreted by archaeologists as calcite are frequently grains of quartz… It is easy to imagine the interpretative confusion which can arise if only quartz is noted at one site and only calcite at another.* We totally agree with the assessment made by these authors. As petrographically documented, some pottery samples (e.g. TSB-17/23 and TSB-7/159) rich in subrounded to subangular monocrystalline quartz (20-30%) up to 600 μm in length, have shown under the binocular microscope translucent crystals that are very similar to those observed in pottery samples tempered with spathic calcite(Figs. 3a and 3b).

In this regard, the confusion between different kinds of tempers or inclusions can distort a correct classification of the ceramic assemblages and have fatal consequences when studying the emergence, maintenance, distribution and disappearance of technological recipes. However, as these scholars noted, the auxiliary methods tested in our study permit to correct this deflection. In this sense, quartz-rich samples have clearly shown - unlike those enriched with spathic calcite -the lowest percentages and intensity calcite peaks and the highest quartz peak at 3.34 Å in the X-ray diffractograms (Fig. 3c). Furthermore, these pottery samples have also shown much lower CaO amounts, and their composition can be classified as "border calcareous" (i.e., CaO < 5%).

In short, apart from conducting thin sections, XRPD analysis has been proved an effective indicator of the presence of abundant quartz in the ceramic body. This method allows us to state that, most likely, most mineral grains observed in certain samples under a binocular microscope must be related to coarse-grained quartz ceramic bodies and not spathic calcite crystals. Therefore, this approach -in which we extrapolate observations from the features of some pottery samples to a larger number of them - requires some caution and the use of certain auxiliary methods in order to avoid making mistakes that could have important implications in subsequent archaeological interpretations of the results achieved. In any case, it is demonstrated again that the use of chemical or mineralogical methods - which are commonly applied for several purposes in ceramic analysis - is enough to sustain the macroscopic observations and identify the presence of spathic calcite fabrics without

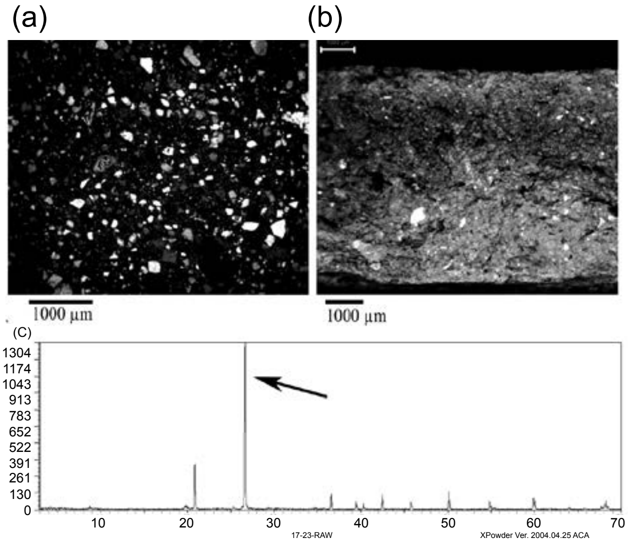

Figure 3: Pottery sample TSB-17/23. A) Microphotograph taken using binocular microscope showing abundant white and translucent coarse particles. Both the appearance and morphology of the crystals are very similar to those observed in the calcite-tempered samples. B) Microphotograph taken using a petrographic microscope, showing 30% of monocrystalline subangular quartz crystals (XPL). C) XRPD diffractogram showing an intense quartz peak at 3.34 Å and the absence of the calcite peak at 3.03 Å.
[Figura 3: Amostra da cerâmica TSB-17/23. A) Fotomicrografia com microscópio binocular mostrando abundantes partículas grossas brancas e translúcidas. Ambas aparência e forma dos cristais são similares àquelas observadas nas amostras de calcita temperada. B) Fotomicrografia com microscópio petrográfico, mostrando 30% de cristais de quartzo subangular monocristalino (XPL). C) Difratograma de raios X mostrando o pico intenso do quartzo em 3,34 Å e a ausência do pico da calcita em 3,03 Å.]

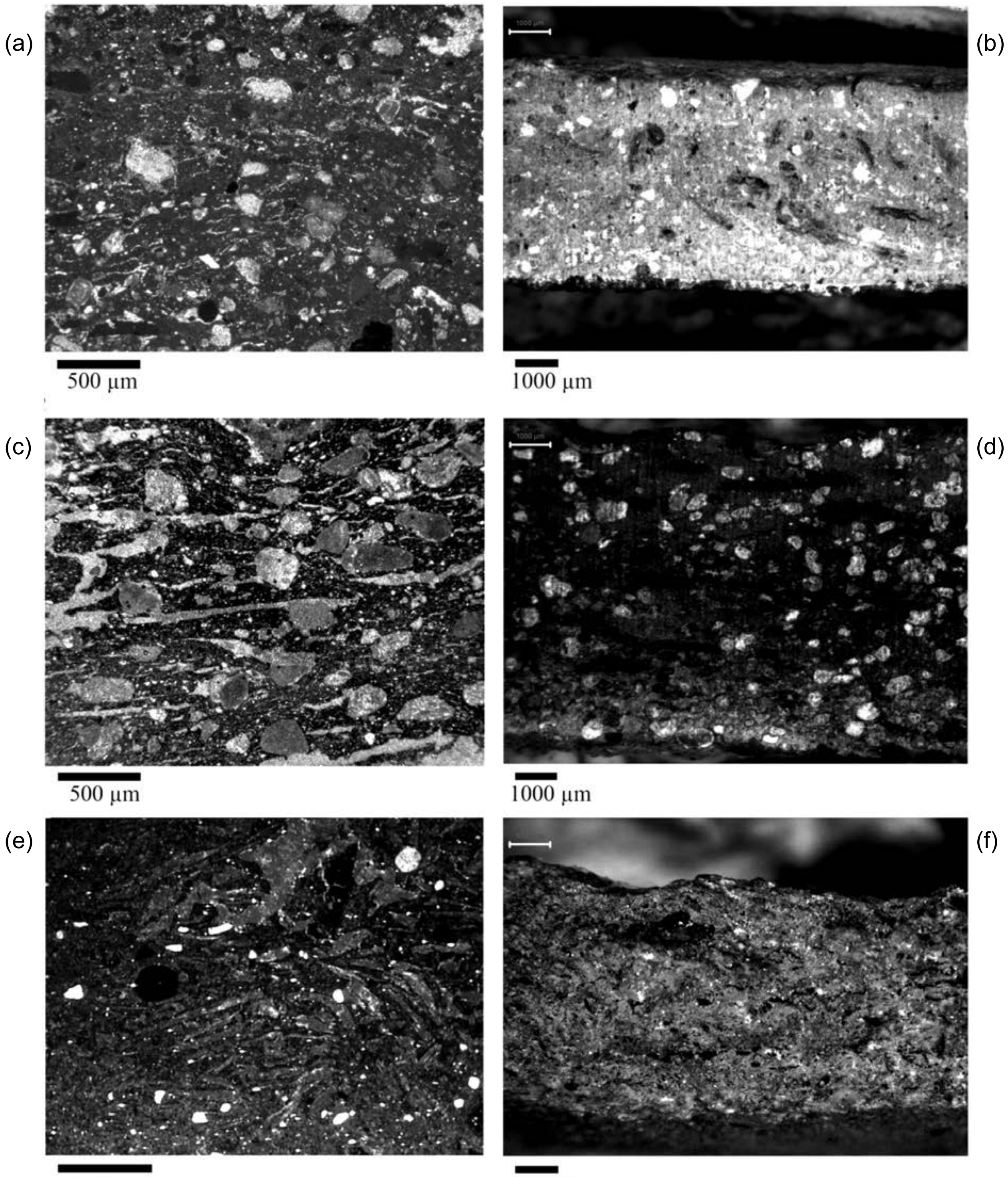

Figure 4: Microphotographs showing the appearance of calcarenite rock fragments in sample SM-181. A) using petrographic microscope (PPL) and B) binocular microscope. Microphotographs showing the appearance of limestone rock fragments in sample SM-140, C) under petrographic microscope (XPL) and D) binocular microscope. Both cases lack translucent grains, note also their rounded morphology and grayish coloration distinct to the particles recorded in samples tempered with spathic calcite. Microphotographs showing the appearance of the untempered, fine-textured and border calcareous sample TSB-6/72, E) under thin section (XPL) and F) binocular microscope.

[Figura 4: Fotomicrografia mostrando o aparecimento de fragmentos de rocha calcarenita na amostra SM-181. A) usando microscópio petrográfico (PPL) e B) microscópio binocular. Fotomicrografias mostando o aparecimento de fragmento de rocha calcárea na amostra SM-140, C) sob microscópio petrográfico (XPL) e D) microscópio binocular. Em ambos casos faltam grãos translúcidos, note também a forma arredondada e a coloração cinzenta diferente da das partículas obtidas em fotomicrografias mostrando o aparecimento de amostras calcáreas não endurecidas de textura fina TSB-6/72), E) sob fina secção (XPL) e F) microscopia binocular.]

the need of thin-sections.

Another limitation when directly extrapolating the features observed using a petrographic microscope to those seen under binocular microscope refers to the detection of sedimentary fabrics showing other calcareous rocks as temper such as crushed limestone, breccias orcalcarenites, among other. These fabrics - when observed under the binocular microscope - may look similar in texture and features to those tempered with spathic calcite (Fig. 4a to 4d). Furthermore, these pastes, given their carbonated composition, also show significant calcite and CaO concentrations in the XRPD and XRF analyses. These characteristics make their identification difficult, even when the non-petrographic auxiliary methods suggested before are used. Nevertheless, the careful examination of the morphology and appearance of the grains of these samples under the binocular microscope reveals that they are slightly different. Thus, in this kind of samples, the crystals are neither translucent nor angular, and they have no well-defined planes.

While there is a distinct habit in both kinds of temper to the trained eye, there may be some confusion when distinguishing them. Since the color of the grains is usually similar due to the same mineral composition, there exists a tendency to classify these calcareous ceramic fabrics alongside those associated with crushed spathic calcite crystals. However, the presence of these calcareous tempers is rare in our samples, and their inclusion within the spathic calcite fabric may be even advisable in technological and taxonomical terms. Saving the perceptual differences related to the use of different types of rock and the exploitation of diverse raw materials, all these technological choices have a common denominator. In other words, though there are slight differences in the properties of the pottery produced depending on the micriticor spathic nature of the calcite [28], potters consciously or unconsciously added in all cases tempers of a carbonated nature. In this sense, a large group encompassing all these different calcareous fabrics would be acceptable and even desirable in archaeological and taxonomic terms as this - with few differences - brings together pottery vessels associated with similar technologies.

In any case, it should be emphasized that discrimination between spathic calcite recipe and those characterized by the addition of crushed limestone or other calcareous rocks is more committed and problematic using binocular microscopy. Distinguishing between fabrics formed by these different components usually requires performing petrographic analysis in order to obtain reliable and conclusive results. Since they are formed with the same mineral and chemical composition, conclusive results to differentiate one material from another cannot be obtained using auxiliary methods such as XRPD and XRF.

After clarifying the limitations found between the correspondence of the petrographic microscope observations and those made with binocular microscope, one can conclude that the latter optical method can be used with certain guarantees of success to differentiate between samples with abundant spathic calcite temper and those that do not possess or have it in very low concentrations (Fig. 4e and 4f).Thus, fabrics with abundant spathic calcite or other calcareous rocks are clearly opposite in texture to those that show a very fine grain-size and have only few mineral inclusions. The latter fabrics did not show - when examined under thin section - the presence of intentionally added mineral as temper. As with coarse quartz-rich fabrics, pottery samples with fine textures also have low % of calcite and CaO in XRPD and XRF analyses, respectively. Moreover, observations made by means of binocular microscopy demonstrate that when the fabric is fine textured and no mineral temper was added, there is a total absence of large and translucent spathic calcite crystals. Thus, examining the ceramic assemblage using a binocular microscope can allow us to potentially distinguish between tempered and untempered pottery vessels and, therefore, among technologies and products related to different properties.

CONCLUSIONS

The main objective of this article was to evaluate the potential of several methods - especially those more accessible to researchers such as binocular microscopy - to identify ceramic fabrics closely related to the spathic calcite recipe. In this sense, the optical examination of ceramic bodies by means of binocular microscope combined with image analysis has allowed the establishment of some optical features and textural groupings which are directly related to certain technological choices made by potters. The data acquired using this method have been tested and related to different archaeometric results obtained from diverse methods, including petrographic analysis, X-ray powder diffraction (XRPD) and X-ray fluorescence (XRF). Correspondence between optical, textural, petrographic, chemical and mineralogical data was observed among the different samples. After exposing the possibilities and limitations of the different methods, we found that an initial classification of the ceramic record by means of binocular microscopy - when combined with chemical (e.g. XRF) or mineralogical analysis (e.g. XRPD) - is feasible. The several tests carried out demonstrated certain reliability of binocular microscopy in extrapolating the identification of spathic calcite recipe to a larger series of ceramics from the analysis of the coarse fraction of the paste. In this sense, the combination of these methods allows a significant increase in the knowledge about the spatial and chronological distribution of this pottery recipe without requiring thin-sections and petrographic observations. Hence, this procedure is particularly useful for analyzing large series of ceramic materials, as well as in the study of pottery vessels that cannot be sectioned for petrographic analysis. Obviously, this approach is not without problems, since it has shown a number of cases linked to the addition of other calcareous rocks that necessarily required using petrology to discern the kind of technological choice that was followed by the potters. However, it is feasible to undertake a reliable classification of ceramic assemblages on the basis of technological criteria and a preliminary study of the emergence, distribution, maintenance or rejection of the spathic calcite recipe by combining certain methods that are quite accessible to most researchers. The combination of these methods permit, among other aspects, to record the existence of variations in the quantity of crushed spathic calcite used in a given period or site, as well as studying its relationship with specific pottery types. In this sense, it should be highlighted that the methodological approach undertaken is decisive, since the identification and characterization of crushed calcite recipe and its distribution in wide Western Mediterranean regions are crucial in archaeological terms. Among other aspects, they can give evidence of cultural contacts and social interactions, the technological cohesion of potters and how knowledge transmission systems and learning strategies were structured within a Mediterranean cultural *koine*.

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