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An assay on simulation of the late Roman amphoric ceramic raw materials

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Abstract

A statistical study of the physico-chemical and mineralogical properties have been carried out on a select group of the late Roman amphora from the Museo Municipal of Ceuta, Spain, and on several clay seams potentially used in ancient times and located in the surroundings of Ceuta (on the African coast of the Straits of Gibraltar), has offered us the possibility of finding analogies between them. The analysis performed enabled us to link, as a first approach, the amphora with the clay materials used in their manufacture, by mainly using factors involving iron oxide content and the type of feldspar. © 1998 Elsevier Science B.V.

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1. Introduction

Nowadays we have much documentation relative to the findings of industrial complexes active during the late Roman Empire at Ceuta, devoted to the processing and commercialization of salt-preserved fishing products. The existence of these factories creates the necessity of having amphorae in order to can the locally made preserves. Recent field works have contributed to the knowledge of the period of activity of these factories in old *Mauretania Tingitana* [1] whose existence is known at least from the I–II centuries AD [2–4]. The amphora type used during this period of the late Roman Empire (from the middle of the III to the V century AD) in the Straits of Gibraltar for the com-

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mercialization of the salted fish products is mainly the Keay XIX amphora [5–7].

The evidence of many amphorae of the type mentioned above in many of these industries at Ceuta makes us think about the use of this container for the salted fish produced here. Thus, the possibility of the manufacture of these amphorae in Ceuta, old *Septem Frates*, or in the nearthy geographical environment is strong. In view of the absence of kilns sited in *Mauretania Tingitana*, analytical work has been carried out in order to establish this hypothesis. The aim of this work has been to use the mineralogical and chemical characterization of some amphorae and clays from Ceuta in order to establish the possible clay sources used in the manufacture of these Roman artefacts, and also to verify the possibility of a local manufacturing, like the archaeological traces makes us think.

The clay samples were selected according to three criteria.

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The finding of ceramic workshops corresponding to other historical periods that could also have worked in Roman times.

The search of modern kilns that perpetuated a craft activity from ancient times.

Lastly, choosing some clay-supplying seams, possibly used in Antiquity because of the strength of the clay, and the proximity of water sources.

2. Materials and methods

Two kinds of materials have been employed: amphora fragments and raw material from different clay seams.

Altogether, 19 amphora samples were analysed, some from the Museo Municipal of Ceuta [8] and all of them having been recovered in underwater contexts, consisting in Keay XIX amphora fragments (Fig. 1).

The second kind of materials employed due to the scope of our work were nine clay samples from oldquarry seams, situated in the vicinity of Ceuta, which had been used as raw materials for the supply of clay to ceramic workshops in different times. These three chosen sites are the following: the Llano de las Damas, LLD, (3 samples), placed in the modern city centre; the Tejar de Ingenieros, TI, (2 samples) and old ceramic workshops [9]; and the so-called Fabrica de Ladrillos, FL, (4 samples), that has been working till some years ago.

The following determinations have been applied to all the samples:

(a) Mineralogical analysis by X-ray diffraction using a Philips PW-1035 diffractometer, working with a copper anticathode and a nickel filter [10].
(b) Chemical analysis [11] determining the major and minor elements by atomic absorption spectroscopy, using a Perkin–Elmer 503 spectrometer, excepting Na and K, performed by flame emission.
(c) Thin sectioning of the samples for observation in a petrographic polarization Orto Plan Pol Leitz microscope. The clay samples have allowed us their hand processing and heating from 500° to 950°C, enabling us afterwards to carry out study of thin sections obtained in this thermal treatment.

(d) Dilatometric study CRH on a cylinder, once the

sample was molten and compressed (100 MPa), with alumine patron, in the dilatometer Adamel Lhomargy Di-24 of silicon carbide furnace and LVDT [12].

3. Results

The mineralogical composition of the ceramic clays from the veins (Table 1) presents some differences, specially the ones from the Llano de las Damas, which contain a higher proportion of phyllosilicates, with 19% of kaolinite and a lesser proportion of quartz, 25%, in their proportions. The samples from the Tejar de Ingenieros present the lesser proportion of phyllosilicates, specially noticeable in them being the presence of smectite, 17%, and the higher quartz proportions, 51%. The Fabrica de Ladrillos samples show an average composition of quartz and phyllosilicates.

Comparing the mineralogical and chemical analyses of the amphorae with the ones of the clay-seam samples, we can notice the presence of a higher proportion of calcite in the amphoric fragments, due, possibly, to the use of a slip in the manufacture of these Roman vessels. The oligoelements tested show different results (Table 2). The copper presents a more or less similar concentration in all samples of amphorae fragments and clays seams. The lead is a big higher in the amphorae, and in these the presence of zinc vessels is specially noticeable.

On account of the results obtained, and in trying to determine possible relations among them, a statistical processing of the data was carried out, using the BMDP 4M [13] program with 22 variables of mineralogical and chemical nature, corresponding to the materials of the clay-seams samples.

In the same way, a similar multivariant factor analysis was employed with the amphorae fragments, including 18 variables.

In the third place, we also applied a similar statistical treatment joining the amphoric samples and the ones of the clay seams, using 18 variables.

After the processing of the data according to the variables considered at the beginning, some new ones were obtained at last, called factors, which explain, respectively, 96.77, 83.37 and 79.29% of the whole variance.



Fig. 1. Late Roman amphora of Keay XIX type.

Table 1				
Mineralogical compositi	on of samples,	, the concentrations	are in	%

Sample	Calcite	Quartz	Dolomite	Feld. K	F. CaNa	Phyllos.	Gypsum
268	15	24	<1	3	_	58	_
269	4	59	_	6	<1	28	3
270	35	37	2	7	_	18	1
271	21	22	1	_	2	51	3
272	28	32	5	-	12	23	-
273	7	42	3	23	_	22	3
274	29	20	4	-	19	16	12
276	31	37	5	-	5	22	_
277	30	37	6	3	6	18	_
278	19	27	_	_	4	42	8
279	47	16	3	9	_	25	_
280	20	33	2	22	_	13	10
281	10	42	_	18	_	30	_
282	12	25	1	3	3	56	_
283	43	35	1	_	7	14	_
284	25	39	1	_	_	35	_
285	15	37	-	-	11	37	_
287	6	28	3	3	2	58	_
288	8	68	1	-	2	21	_
LLD	-	25	-	6	_	69	_
FL	-	36	-	10	-	54	_
TI	2	51	1	<1	-	46	-

Table 2 Chemical composition of samples, major elements are in percentage, minor elements in ppm

Sample	SiO ₂	Al_2O_3	CaO	MgO	K ₂ O	Na ₂ O	Fe ₂ O ₃	MnO	TiO ₂	Cu	Pb	Zn
268	52.71	17.91	5.35	8.12	9.00	3.20	3.87	0.31	0.56	19	104	165
269	44.92	17.33	4.27	4.93	5.76	16.22	5.90	0.05	0.62	31	129	420
270	50.03	17.84	3.60	3.82	4.53	12.17	5.40	0.05	0.56	27	183	407
271	62.47	16.33	1.08	0.11	4.93	9.98	4.98	0.07	0.48	36	99	227
272	51.14	15.08	3.57	0.12	6.27	17.83	5.58	0.08	0.33	30	95	266
273	68.66	16.34	3.77	< 0.01	4.75	0.34	5.70	0.06	0.38	27	39	160
274	73.36	16.00	2.88	< 0.01	2.52	0.68	4.33	0.08	0.15	35	59	100
276	68.48	15.62	0.87	6.01	3.20	3.87	1.42	0.07	0.46	29	57	542
277	59.01	16.71	4.83	2.59	4.18	9.42	2.67	0.08	0.51	21	6	661
278	46.28	16.32	1.53	0.90	5.40	22.38	6.79	0.05	0.35	40	104	259
279	55.62	16.46	2.82	3.72	5.06	11.53	4.14	0.07	0.58	35	<1	110
280	69.98	18.14	1.85	0.11	4.44	0.21	4.73	0.06	0.51	30	56	197
281	64.02	17.20	3.69	0.38	1.38	15.41	7.38	0.08	0.46	63	<1	120
282	71.44	16.28	0.73	0.05	4.73	0.44	5.82	0.05	0.46	28	99	476
283	74.78	15.51	2.66	< 0.01	2.61	0.81	3.25	0.03	0.35	31	45	121
284	54.67	16.90	7.09	0.08	4.33	12.08	4.29	0.05	0.51	28	51	134
285	47.12	16.59	3.17	1.73	8.39	17.43	5.22	0.05	0.30	37	50	398
287	46.92	15.58	3.90	5.43	4.86	17.20	5.80	0.06	0.20	40	130	437
288	50.98	16.77	2.43	0.15	5.88	17.52	5.57	0.06	0.64	42	100	125
LLD	67.47	16.35	0.31	0.12	6.51	5.15	3.90	0.01	0.20	39	51	102
FL	72.69	12.06	0.03	0.35	7.26	3.89	3.34	0.06	0.32	35	57	97
TI	68.13	16.57	0.04	0.25	10.02	1.47	3.32	0.02	0.22	33	45	62



Fig. 2. Plot of the ceramic clays as a function of F1 and F2 factors.

The results of the factor analysis corresponding to the samples of the clay seams gather the variables depending on the original materials in the two firstcharge factors, F1 and F2, which are more significant because they explain most of the use of a different original material, because each one of them is linked to a different kind of clay, as for instance, kaolinite and illite.

The graphical representation of the clay-seam samples, according to the two above-mentioned factors (Fig. 2) allow us distinguish the kind of materials observing that the samples from the Llano de las Damas and from the Tejar de Ingeneiros, although showing analogies in their respective clay minerals can be distinguished by the presence of quartz, specially observed in the Tejar de Ingenieros samples. The materials from the Fábrica de Ladrillos are distinguished from the former by the predominance of illite as a clay mineral. These results reveal that, of all samples taken into account, the clays from the Llano de las Damas are the more suitable ones for use as raw material in making vessels, because they present higher proportions of kaolinite, and lower proportions of quartz.

The statistical programme applied to the amphora samples allows us recognise seven charge factors. Factor 1 could be considered as an indicator of the original material, because it groups the amphorae in terms of the kind of feldspar. Factor 2 has, as a variable, the higher charge of iron oxides as opposed to calcite, it being possible to consider them in terms



Fig. 3. Plot of the amphoric materials as a function of F1 and F2 factors.

of the iron oxides as a factor contributing to the original material. The variations of calcite have probably been produced during the manufacturing process. Factor 3 is represented by all the major elements analyzed. This last factor cannot be considered as an indicator of the original material, since the prolonged permanence of our amphorae in the sea has, perhaps, brought about variations in their concentration.

The graphical representation of the amphorae in terms of the first two factors (Fig. 3) classifies the amphorae according to the kind of feldspar and the predominance of iron oxides in their composition. On account of the statistical behaviour that the amphorae samples present, considering these two factors, and because these two factors are the ones that explain the higher percentage of variance, they cannot be used as a criterion that will allow to identify the differences in the raw materials as the variables that represent them are carbonates and the major elements, respectively. Thus, factors 3 and 5 (the iron oxides and the feldspars, respectively) are the ones that can be taken as indicative of the genetic material, because the existence of these elements is not modified with the thermal treatment that the amphorae suffered during their firing, neither are they affected by the constant immersion condition in the sea due to the archaeological context of the finding.

In Fig. 4, we represent the distribution of the amphorae and of the clay-seam samples in terms of



Fig. 4. Plot of the ceramic clays and the amphorae as a function of F3 and F5 factors.

these two factors: we can notice the separation of the clay-vein samples, and the association of some of the amphorae with each one of the seams taken into account.

Corroborating our hypothesis, we have also found coincidences between the thin sections of the Keay XIX amphorae and the simulated pastes made by us with Llano de las Damas clays (Fig. 5). The dilatometric curves $T(^{\circ}C)\Delta I/I_0$ shows that the material undergoes a slight shrinking up to 750°C, when sintering begins. From the curves $d(\Delta I/I_0)$ vs. T (Fig. 6), for the amphorae and the sample of the Llano de las Damas, the α/β phase transition of quartz can be observed, selon in the cooling curve at 800°C. The possible materials were transformed and their changes on account of dilatation or contraction process are not reversible. The carbonates do not show the effects of decomposition in the oxides.

4. Concluding remarks

The mineralogical and chemical data analyzed here let us establish similarities between the clay used in making the amphora and those of the three seams from the region of Ceuta (Llano de las Dams, Tejar de Ingeneiros and Fábrica de Ladrillos) taken into account in this work.

The feldspar and the iron oxides are the entities that let us make a typification. We propose, therefore, that these late Roman Keay XIX amphorae must have been locally manufactured in the surroundings of the city, in workshops that still have not been discovered.



Fig. 5. Photograph of thin section of simulated paste with Llano de las Damas clay; <> thin section of Keay XIX amphora fragment (X32).



Fig. 6. Dialatometric curves of the paste of Llano de las Damas and Keay XIX amphora fragment.

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