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Geochemical signatures of Roman amphorae produced in the Sado River estuary, Lusitania (Western Portugal)

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ABSTRACT

Several archaeological sites located in the Sado estuary, Lusitania province (Western Portugal), show evidence of an important amphorae production, mainly related to the Roman Tróia industrial centre of fish preserves. The chemical analysis of amphorae sherds found in Barrosinha, Bugio, Zambujalinho, Xarrouqueira and Vale da Cepa (Lower Sado) was performed by instrumental neutron activation analysis, and the mineralogical composition obtained by X-ray diffraction. The chemical results were compared with amphorae from the Herdade do Pinheiro production centre, also located in the Sado estuary. Application of several exploratory and discriminant techniques of multivariate statistical analysis using chemical data of Lower Sado amphorae allowed the characterization and differentiation of two reference groups, named Sado 1 and Sado 2. The geochemical patterns and the mineralogical composition of these groups indicate the use of two different raw materials, the selection of which appears to have been based on the proximity factor of the kilns. The amphorae produced in the Sado basin were differentiated from amphorae from the Gualdalquivir estuary (Villa Nueva, Cádiz, Spain) in Baetica province.

1. Introduction

The characterization of Roman amphorae produced in the different sites/regions is essential to establish the importance and relevance of each site in the Roman economy and society in general.

Some of the most important productions centres in the Lusitania province were located in the estuaries of Sado and Tagus, the amphorae produced in these sites being mainly used in storage and shipping of fish-based products (Diogo and Faria, 1990; Étienne et al., 1994; Mayet et al., 1996; Mayet and Silva, 2002; Fabião, 2004). According to Mayet et al. (1996), amphorae produced in the Baetica province in the late period (from the third century onwards) may also have been exported as empty vessels to be used in the fish-sauce industries of Lusitania.

Estuarine sediments tend to be very homogeneous in composition due to natural geological processes of homogenization during transport and deposition of the sediments. Therefore ceramics produced using sediments of a particular estuary may be not easily distinguishable. Thus a deep analysis of the paste composition, together with archaeological evidence, is required to determine the subtle but significant differences used as indicators to establish provenance.

Previous archaeometric studies of amphorae from three important production centres of the estuaries of both rivers – Porto dos Cacos and Quinta do Rouxinol in Tagus, and Herdade do Pinheiro in Sado – have been performed in order to establish the chemical characterization of the amphorae produced in these centres (Cabral and Gouveia, 1984; Cabral et al., 1993–1994, 1996, 2000, 2002; Prudêncio et al., 2003; Dias and Prudêncio, 2007). Those works, based on the instrumental neutron activation analysis (INAA), have shown that distinguishing amphorae produced in these sites may not be obvious, due to the use of similar raw materials belonging to the Tertiary Tagus-Sado basin. Later on, geochemical considerations and statistical treatments were used in order to determine subtle differences among the three production centres (Prudêncio et al., 2003; Raposo et al., 2005; Dias and Prudêncio, 2007).

Archaeological surveys have shown the existence in the Lower Sado of other production centres, besides Herdade do Pinheiro; the amphorae produced in these units were used in the export of fish preserves, of which Tróia was one of the most important centres. In fact an intense amphorae production occurred in the Sado estuary possibly since the period of Augustus and Tiberius, and mass production was already in place by the time of Claudius and continued up to the mid-fifth century AD (Mayet et al., 1996). The location of the kilns is certainly related to the proximity of the river, and modifications of the river dynamics during the long period of amphorae production (500 years) may explain the move/spread of their locations (Fabião, 2004).

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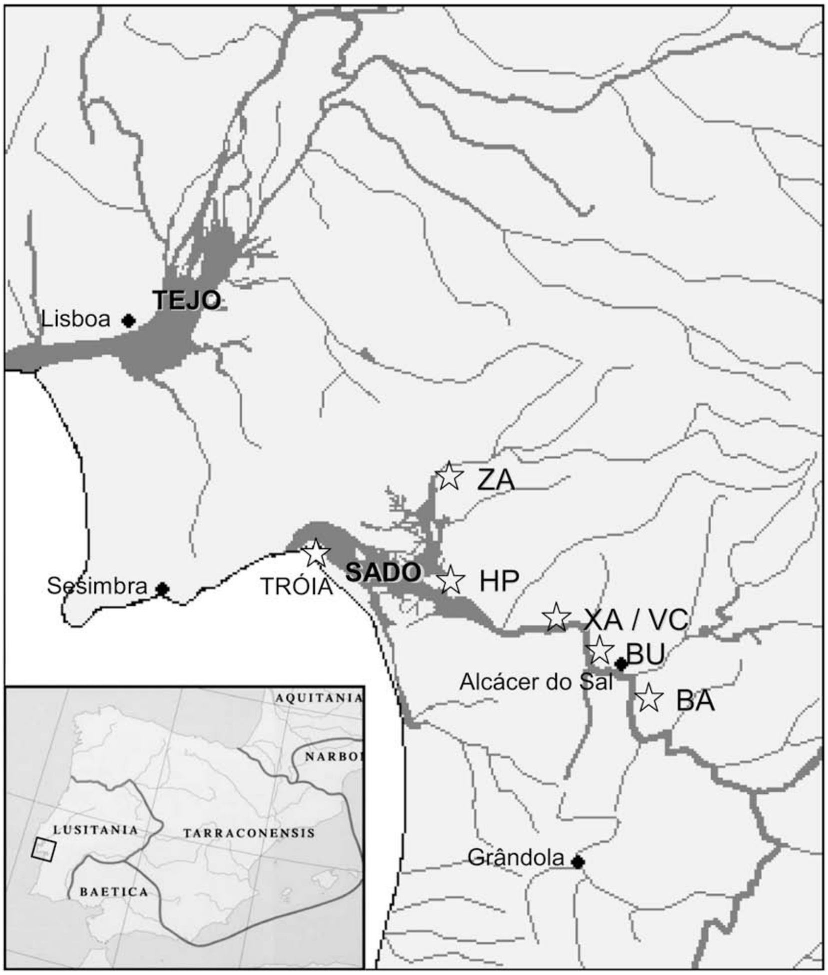

Fig. 1. Map of the Lower Sado with the archaeological sites studied and mentioned in the text (BA – Barrosinha, BU – Bugio, ZA – Zambujalinho, XA – Xarrouqueira, VC – Vale da Cepa, HP – Herdade do Pinheiro).

In the present work amphorae from five different archaeological sites of the Lower Sado, namely Barrosinha, Bugio, Zambujalinho, Xarrouqueira and Vale da Cepa, are studied. Chemical analyses of the amphorae sherds were performed by INAA at ITN using the same analytical protocol as for Herdade do Pinheiro (Cabral et al., 2000). This was done in order to obtain a more complete chemical signature of amphorae produced in the Sado estuary. Multivariate statistical analysis of the chemical data obtained so far for the six archaeological sites (Herdade do Pinheiro included) was performed, using non-supervised and supervised methods in order to characterize and differentiate eventual groups. Mineralogical analysis of selected sherds was done by X-ray diffraction (XRD) in order to complement the chemical composition. The results are discussed taking into account geochemical and mineralogical considerations, and the geological context.

The chemical composition of the Sado productions is compared with other estuarine amphorae-producing areas located in the Baetica province, particularly amphorae from the Guadalquivir River estuary, Villa Nueva, Cádiz, Spain (Dias et al., 2003), in order to establish the respective differences.

Thus the characterization of ceramic productions in different units of the Lower Sado, with the establishment of compositional reference groups' of the amphorae produced in this region, is the major goal of this work, which is fundamental for the further pursuit of studies concerning amphorae distribution and exchange during Roman times.

Table 1
Radionuclides, cooling times, energies of γ rays, and type of detector used.

Element	Radionuclide	Decay time (d)	Energy of γ rays used (keV)	Detector Coaxial Ge(Li)	LEPD
Na	^{24}Na	3	1368	x	
K	^{42}K	3	1524	x	
Sc	^{46}Sc	27	889	x	
Cr	^{51}Cr	27	319	x	x
Fe	^{59}Fe	27	192; 1099	x	x
Co	^{60}Co	27	1332	x	
Zn	^{65}Zn	27	1115	x	
As	^{76}As	3	559	x	
Rb	^{86}Rb	27	1077	x	
Sb	^{124}Sb	27	1690	x	
Cs	^{134}Cs	27	796	x	
Ba	^{131}Ba	27	496	x	x
La	^{140}La	3	1596	x	
Ce	^{141}Ce	27	145	x	x
Nd	^{147}Nd	27	91; 531	x	x
Sm	^{153}Sm	3	103	x	x
Eu	^{152}Eu	27	1408	x	x
Tb	^{160}Tb	27	881; 1178	x	x
Yb	^{169}Yb	27	177; 198	x	x
Lu	^{177}Lu	27	208	x	x
Hf	^{181}Hf	27	482	x	
Ta	^{182}Ta	27	1222	x	x
Th	^{233}Th	27	300; 312	x	x
U	^{239}Np	3	106; 228	x	x

Table 2

Chemical results obtained by INAA for amphorae sherds of Barrosinha, Bugio, Zambujalinho, Xarrouqueira and Vale da Cepa (μg/g unless expressed otherwise). D14 = Dressel 14; D14b = Dressel 14b; D30 = Dressel 30; A50 = Almagro 50; A51c = Almagro 51c; A51a–b = Almagro 51a–b; ind. = indeterminate typology.

Sample	Type	Na_2O	K_2O	Fe_2O_3T	Sc	Cr	Co	Zn	As	Rb	Sb	Cs	Ba	La	Ce	Nd	Sm	Eu	Tb	Yb	Lu	Hf	Ta	Th	U
BA-1	D14	0.890	2.18	5.15	17.7	78.2	12.3	99.0	8.10	110	0.956	5.87	394	41.8	80.6	40.0	7.60	1.61	1.21	3.82	0.571	6.87	1.37	13.2	2.77
BA-2	D14	0.840	2.08	5.76	16.4	77.2	11.9	64.3	6.18	104	0.963	5.69	397	35.1	66.4	32.5	6.24	1.35	0.885	3.32	0.502	7.04	1.27	11.8	3.06
BA-3	D14	0.976	2.26	4.74	17.2	74.9	14.7	67.5	5.20	109	1.02	5.73	398	42.7	80.7	41.6	8.19	1.68	1.13	3.69	0.56	6.75	1.37	12.4	3.02
BA-4	D14	1.250	2.36	5.46	17.1	74.2	16.5	73.6	11.4	117	0.823	5.73	454	42.0	81.5	40.5	7.85	1.58	1.23	3.85	0.589	7.86	1.39	12.9	2.45
BA-5	D14	0.755	2.11	4.91	15.5	65.1	16.2	71.9	5.64	99.6	1.01	5.14	338	35.3	69.9	29.6	6.35	1.38	1.04	3.4	0.524	7.43	1.27	12	2.78
BA-6	D14	0.819	1.94	4.75	15.0	66.2	8.41	60.1	7.86	92.3	1.07	4.81	317	32.6	66.3	32.4	5.84	1.31	0.938	3.2	0.486	7.11	1.31	11.5	3.23
BA-7	D14	1.30	2.13	3.99	14.5	67.3	12.3	135	4.76	97.3	0.897	4.63	378	36.6	72.2	35.0	6.45	1.37	0.814	2.94	0.447	6.67	1.27	11.1	2.07
BA-8	D14	1.41	2.26	5.34	15.8	66.5	16.2	68.8	10.1	109	0.812	5.56	455	41.8	81.2	41.4	7.89	1.64	1.09	3.8	0.564	7.55	1.34	12.1	2.53
BA-9	D14	1.32	2.36	5.17	17.3	80.2	14.0	63.5	4.96	109	1.06	5.89	380	43.6	82.7	41.1	8.04	1.76	1.08	3.68	0.55	7.3	1.37	12.4	3.16
BA-10	D14	1.30	2.27	5.20	17.2	78.1	15.1	77.3	4.77	111	0.911	5.79	346	39.8	75.9	39.1	7.49	1.66	1.06	3.52	0.53	7.19	1.24	11.8	2.87
BA-11	D14	1.17	2.27	4.72	14.9	62.3	13.4	66.8	7.06	107	0.744	5.31	416	38.1	74.3	35.7	6.97	1.42	1.09	3.71	0.565	8.12	1.29	11.8	1.78
BA-12	D14	0.871	2.21	4.85	15.1	69.2	13.3	65.3	7.28	99.4	0.852	5.3	391	40.2	75.9	36.5	7.64	1.63	1.11	3.43	0.524	7.02	1.19	11.5	3.04
BA-13	D14	0.792	2.09	4.64	14.3	63.0	9.10	66.7	8.39	99.4	0.779	5.21	311	34.9	69.4	34.0	6.31	1.29	0.922	3.00	0.527	6.13	1.21	10.7	2.89
BA-14	D14	0.345	3.09	4.88	12.9	59.6	10.1	75.1	14.1	183	0.611	11.9	375	36.3	72.9	35.9	6.73	1.25	0.874	2.81	0.44	5.83	1.63	14	3.98
BA-15	D14	1.54	2.21	5.14	15.2	58.3	14.7	72.2	11.7	109	0.829	5.29	386	42.0	85.0	41.6	8.13	1.55	1.18	3.79	0.615	7.46	1.31	11.8	3.49
BA-17	D14	0.882	2.17	4.56	15.6	67.3	9.28	65.9	6.85	97.6	0.939	5.15	348	38.0	73.5	32.9	6.94	1.35	1.16	3.38	0.543	7.28	1.22	12.1	2.61
BA-18	D14	1.26	2.39	5.28	16.7	65.0	17.8	88.6	9.95	118	0.792	5.83	406	43.6	93.1	40.9	8.13	1.64	1.32	4.06	0.607	7.65	1.45	13.3	2.95
BA-19	D14	1.23	2.04	4.34	12.6	50.8	12.6	65.8	9.25	90.8	0.81	4.4	346	35.7	69.3	33.4	6.72	1.33	0.971	3.16	0.526	6.49	1.06	10.7	2.55
BU-1	D14b	0.51	2.08	5.60	15.4	62.7	9.79	61.0	6.24	101	0.787	5.52	367	31.1	63.0	30.4	4.72	1.28	0.853	2.95	0.489	5.85	1.19	10.5	2.94
BU-3	D14b	0.624	1.98	7.46	17.4	94.8	17.0	60.0	13.4	100	1.16	5.08	328	35.2	79.4	37.0	6.29	1.46	1.01	3.3	0.521	6.64	1.4	12.1	3.81
BU-4	D14b	1.03	2.36	6.04	16.7	72.0	14.8	80.8	9.12	118	0.899	6.25	408	40.6	72.4	37.8	7.24	1.56	1.2	3.79	0.553	6.77	1.32	12.2	3.17
BU-6	D14b	1.29	2.23	6.31	16.9	75.4	17.9	77.3	9.23	109	0.983	5.68	402	38.2	74.1	37.4	7.26	1.44	1.07	4.64	0.639	6.59	1.29	12.1	4.63
BU-7	D14b	0.657	2.29	5.78	14.9	104	11.7	75.5	7.42	94.3	0.523	5.96	464	36.2	72.2	33.0	5.84	1.35	0.85	2.8	0.429	6.06	1.28	10.8	3.3
BU-8	D14b	1.05	2.4	5.44	16.5	76.1	11.4	61.8	10.4	104	0.840	5.08	454	38.4	72.8	35.2	6.75	1.41	0.96	3.54	0.553	7.41	1.36	13.6	3.03
BU-9	D14b	0.749	2.3	6.09	18.7	78.8	15.2	61.4	9.29	111	1.17	6.2	437	41.6	81.0	35.0	7.61	1.59	1.08	3.77	0.573	7.2	1.41	12.7	3.83
BU-10	D14b	1.28	2.29	6.21	17.8	80.5	15.4	69.6	11.0	108	1.08	5.47	372	38.1	70.9	31.3	6.94	1.5	1.04	3.8	0.569	8.38	1.45	13.2	4.53
BU-11	D14b	0.675	2.1	6.07	17.1	75.9	12.2	59.7	7.33	104	0.957	5.52	311	33.5	63.6	31.7	5.93	1.36	0.936	3.19	0.542	6.74	1.31	11.9	4.62
BU-12	D14b	0.761	2.15	5.18	15.0	71.1	9.60	56.0	4.24	100	0.972	5.18	328	32.3	58.9	27.7	5.38	1.1	0.898	3.18	0.51	7.61	1.31	12.2	3.23
BU-13	D14b	1.06	2.24	5.63	16.9	77.0	12.2	67.6	10.5	106	1.03	5.45	454	36.5	72.1	33.1	6.37	1.42	1.06	3.7	0.557	7.61	1.45	13.2	2.99
BU-14	D14b	1.21	2.45	6.09	18.3	79.9	19.1	86.6	2.66	112	0.912	5.72	393	43.0	81.4	36.8	7.69	1.55	1.16	3.73	0.576	6.87	1.49	13	3.69
BU-15	D14b	1.07	2.31	4.99	12.8	57.0	12.3	60.6	7.69	92.9	0.829	4.8	333	30.6	58.0	30.0	5.43	1.18	0.769	3.08	0.462	6.2	1.09	10.4	3.31
BU-16	D14b	0.775	2.1	5.41	15.0	60.9	16.2	63.0	8.06	89.9	0.780	4.77	506	34.1	66.2	29.5	5.97	1.37	0.921	3.01	0.456	6.25	1.13	11.1	2.68
BU-17	D14b	0.442	1.92	5.58	15.6	61.4	7.99	48.5	6.82	81.1	0.687	4.37	1242	25.7	57.4	24.5	4.70	1.01	0.84	2.74	0.417	5.69	1.14	11	2.82
BU-18	D14b	0.814	2.14	4.90	15.8	66.8	15.0	62.5	4.03	106	0.857	5.24	402	33.9	72.0	31.9	6.03	1.28	0.914	3.12	0.472	6.38	1.32	11.3	2.23
BU-20	D14b	1.01	2.44	6.51	19.0	90.5	21.1	75.3	11.0	115	1.06	6.12	435	43.8	96.6	43.0	8.14	1.68	1.25	4.05	0.62	8.1	1.5	14.5	3.08
BU-2	A51c	1.23	2.48	5.31	15.6	83.3	11.1	63.3	18.0	113	1.68	8.11	323	38.6	72.3	35.9	5.98	1.3	0.826	2.58	0.409	6.72	1.2	11.5	3.37
XA-1	D14b	1.08	2.26	4.93	15.4	68.8	17.8	171	7.61	115	0.874	5.51	401	38.9	78.7	37.4	7.83	1.51	1.08	3.32	0.533	6.59	1.18	11.6	3.39
XA-2	D14b	0.559	2.95	4.45	12.7	52.3	9.29	178	14.8	190	0.584	12.9	869	49.2	96.7	50.2	10.3	1.64	1.38	4.22	0.642	9.18	2	19.8	5.27
XA-3	D14b	0.908	2.12	3.95	14.1	63.8	11.9	196	24.4	106	0.892	5.21	471	35.9	72.7	33.0	6.95	1.42	0.896	3.09	0.526	6.92	1.2	10.9	4
XA-4	D14b	0.963	2.18	6.90	12.2	54.4	15.9	253	61.9	101	1.05	5.1	358	32.9	63.6	33.5	7.22	1.37	1.03	2.85	0.495	5.9	1.09	9.69	3.43
XA-6	D14b	0.261	3.03	3.87	11.0	51.6	7.13	51.2	14.3	152	0.508	9.88	451	39.0	74.5	39.2	8.36	1.2	0.975	3.36	0.539	8.92	1.85	17.4	4.85
XA-17	D14b	1.11	2.26	4.88	14.2	60.7	18.8	73.0	13.1	104	0.844	5.28	437	38.5	74.0	36.8	5.97	1.57	1.13	3.56	0.573	6.6	1.22	10.9	3.16
XA-18	D14b	1.01	2.07	3.56	11.2	51.6	13.8	57.4	3.02	95.4	0.751	4.52	416	31.2	63.2	29.9	5.92	1.14	0.83	2.73	0.44	5.79	0.92	9.51	2.6
XA-19	D14b	0.283	3.31	4.26	13.6	59.7	7.52	73.5	10.7	176	0.569	11.7	1027	37.8	72.5	37.9	8.88	1.3	1.08	3.28	0.568	6.74	1.75	16	3.74
XA-20	D14b	0.376	3.2	4.41	13.8	60.9	8.56	75.4	8.74	199	0.574	13.6	522	40.1	80.4	41.9	6.78	1.49	1.17	3.64	0.582	7.18	1.89	17.5	4.56
XA-11	D30?	0.315	3.21	4.50	13.0	57.9	9.56	99.9	11.3	185	0.484	12.1	652	38.4	72.4	37.2	8.18	1.33	1.01	3.2	0.5	6.92	1.82	15.7	3.77
XA-5	A51c	0.752	2.55	3.78	11.3	55.1	9.78	52.8	6.42	151	0.687	8.08	364	31.9	65.5	32.3	4.56	1.07	0.879	2.63	0.412	7.17	1.23	14	3.67
XA-7	A51c	0.68	2.67	4.53	13.2	60.3	10.3	63.7	9.20	167	0.585	10.6	343	34.0	69.9	33.6	5.46	1.18	0.852	2.9	0.437	6.51	1.47	13.8	3.62
XA-9	A51c	0.612	3.36	4.77	13.6	61.0	10.7	75.6	15.6	198	0.729	12.4	526	38.9	74.6	39.4	6.14	1.3	1.02	3.31	0.491	7.21	1.73	16.9	4.2
VC-04	D14b	0.359	3.09	4.30	11.9	51.0	8.10	65.6	11.5	192	0.449	12.1	435	41.3	84.4	42.0	8.07	1.45	1.18	3.68	0.591	8.38	1.89	17.5	3.36
VC-15	D30	0.487	3.13	4.05	14.4	62.9	9.08	88.9	12.3	173	0.581	11.9	436	45.1	85.9	44.6	9.18	1.52	1.32	3.75	0.548	8	1.84	18.2	3.7
VC-02	A51c	0.299	3.06	4.60	11.4	51.3	5.28	62.3	11.1	186	0.433	12.2	368	39.3	79.3	38.9	7.44	1.35	1.04	3.39	0.498	7.91	1.71	17	3.53
VC-17	A51c	0.38	3.37	4.91	14.0	62.9	6.31	68.3	3.18	206	0.660	15.7	342	39.5	76.9	44.5	10.1	2.13	1.38	3.99	0.585	5.54	1.64	15.5	5.16
VC-18	A51c	0.349	3.02	4.25	12.1	53.2	9.42	68.8	8.57	187	0.498	12.1	374	39.6	77.2	38.9	7.60	1.28	1.01	3.31	0.516	7.28	1.67	17	4.03
VC-05	A51a–b	0.327	2.84	3.49	10.9	47.0	7.26	74.0	10.5	185	0.507	11.8	337	38.9	76.9	36.6	7.21	1.18	1.03	3.34	0.522	8.8	1.8	17.3	3.74
VC-06	A50	0.678	2.66	4.58	11.5	71.4	12.0	86.3	9.43	100	0.614	6.27	970	33.4	63.6	28.5	5.91	1.26	0.851	2.72	0.428	6.69	1.09	10.6	2.18
VC-12	A50	0.573	2.38	4.00	10.2	59.1	11.0	67.1	7.40	91.8	0.496	5.47	807	32.3	61.4	29.1	5.56	1.14	0.715	2.42	0.379	6.47	0.983	9.75	1.47
ZA-1	D14	0.88	2.35	3.38	16.8	88.7	13.3	55.4	3.88	138	0.910	6.48	790	32.4	67.9	31.7	6.51	1.29	0.94	3.2	0.49	5.46	1.23	12	8.67
ZA-16	A50	0.42	3.05	3.76	11.3	48.6	8.43	67.0	8.35	158	0.530	9.55	759	42.7	85.3	42.5	7.22	1.26	1.05	3.41	0.49	7.96	1.56	17.2	4.02
ZA-14	A51c	0.62	3.19	4.16	11.1	48.7	8.55	61.8	8.35	157	0.550	9.57	785	45.7	84.8	46.3	8.64	1.52	1.21	3.71	0.54	8.64	1.53	17.2	4.35
ZA-15	A51c	0.46	3.64	4.09	11.0	49.9	11.9	59.5	9.13	185	0.560	10.6	800	40.6	79.7	42.0	9.06	1.36	1.15	3.69	0.55	7.38	1.61	18	4.41
ZA-17	A51c	0.56	3.4	3.57	11.6	49.5	10.9	76.9	7.59	179	0.430	11.1	1111	46.5	91.8	45.4	9.15	1.28	1.14	3.64	0.54	9.14	1.76	20.5	4.65
ZA-7	A51c?	1.23	2.68	3.90	14.4	62.8	18.6	58.8	6.32	128	1.02	5.57	427	41.8	80.3	39.0	7.91	1.57	1.08	3.54	0.55	6.88	1.22	11.7	9.6
ZA-8	A51c?	1.20	2.61	3.81	15.4	74.3	17.9	50.4	5.21	129	0.850	5.92	391	39.2	80.0	37.5	7.49	1.49	1.1	3.59	0.5	6.6	1.3	11.6	17
ZA-9	A51c?	1.11	2.25	5.33	12.7	61.9	18.7	48.8	24.9	115	0.990	5.37	434	31.3	63.4	31.2	6.11	1.19	0.85	2.91	0.43	5.8	1.26	10	3.02
ZA-10	A51c?	1.20	2.65	5.08	14.6	71.6	17.5	62.0	8.53	131	1.03	5.77	486	38.5	75.9	35.9	7.55	1.38	1.01	3.21	0.51	6.79	1.17	11.8	10.5
ZA-11	A51c?	1.00	2.39	4.90	12.6	60.3	19.0	49.8	21.5	127	0.930	5.58	425	32.2	64.4	29.6	6.14	1.13	0.81	2.77	0.41	5.18	1.1	10.5	3.08
ZA-12	A51c?	1.03	2.38	5.26	13.2	62.4	18.7	53.1	19.0	127	0.950	5.60	448	32.4	62.5	32.3	6.25	1.13	0.84	2.84	0.45	5.53	1.11	10.7	3.21
ZA-13	A51c?	0.97	2.35	4.89	12.4	58.7	19.3	48.5	19.5	114	0.850	5.50	440	31.1	63.2	29.1	5.79	1.13	0.83	2.77	0.44	5.22	1.03	9.53	3.07
ZA-18	ind.	0.44	3.26	3.14	10.5	45.3	14.0	72.5	8.50	170	0.380	9.59	1079	44.1	85.5	42.1	8.49	1.31	1.15	3.73	0.5	8.46	1.69	19	4.12
ZA-2	ind.	0.99	2.51	3.23	17.2	80.3	13.1	59.4	5.24	139	0.890	6.98	687	35.0	63.8	31.2	6.57	1.29	0.93	3.07	0.51	5.02	1.26	10.9	9.27
ZA-3	ind.	0.94	2.59	3.39	18.6	86.1	15.3	61.1	4.80	149	0.920	7.36	442	39.6	76.5	36.9	7.08	1.51	1.06	3.43	0.55	5.37	1.25	12	12.1
ZA-4	ind.	1.13	2.29	4.64	14.2	68.0	17.1	43.9	13.2	113	0.950	4.66	973	29.9	63.8	29.3	5.60	1.07	0.82	2.89	0.44	6.31	1.18	11.5	7.26
ZA-5	ind.	0.92	2.54	3.03	16.4	75.1	14.2	53.7	7.92	128	0.810	6.14	817	35.2	70.8	31.6	6.77	1.43	0.97	3.08	0.44	4.72	1.13	10.9	7.22
ZA-6	ind.	1.10	2.44	3.12	17.2	80.8	16.9	65.2	5.12	147	0.820	6.89	454	42.7	86.3	39.5	8.07	1.58	1.15	3.64	0.54	5.84	1.23	11.8	12.8

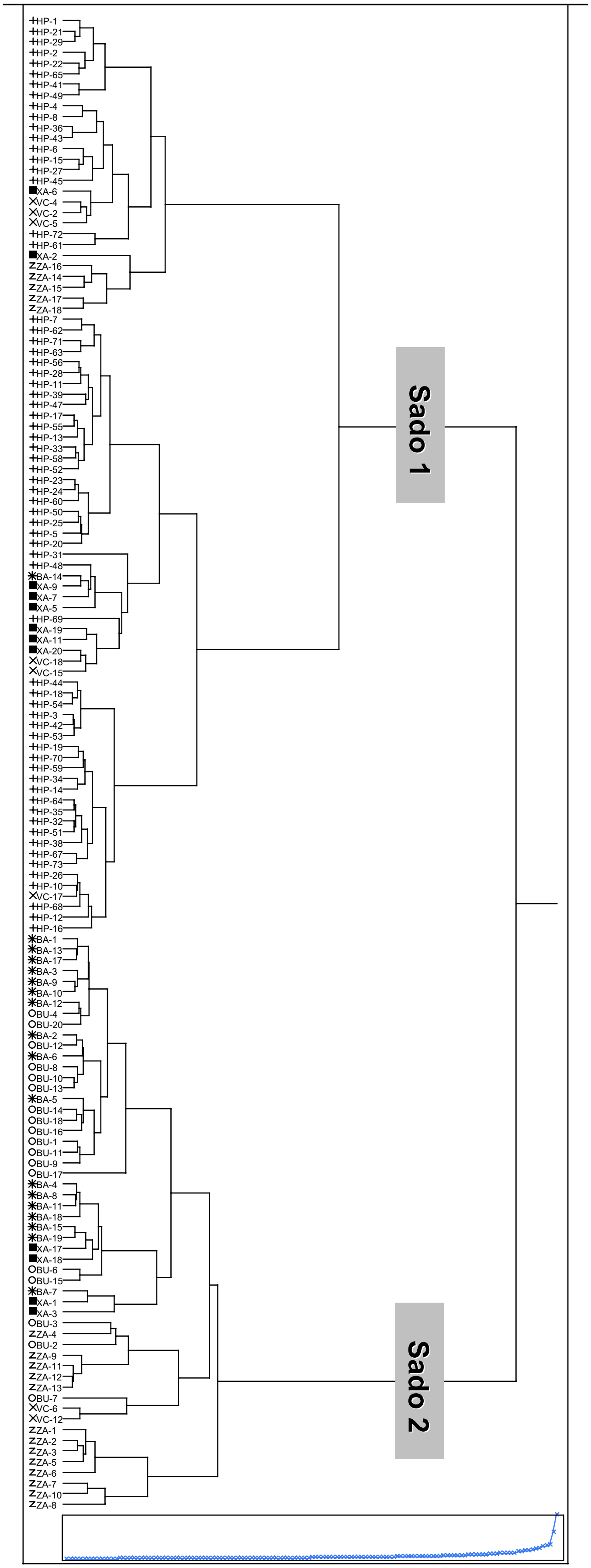

Fig. 2. Dendogram of amphorae samples resulting from the joining-tree clustering using Ward's amalgamation method and the Pearson correlation coefficient (HP data from Cabral et al., 2000).

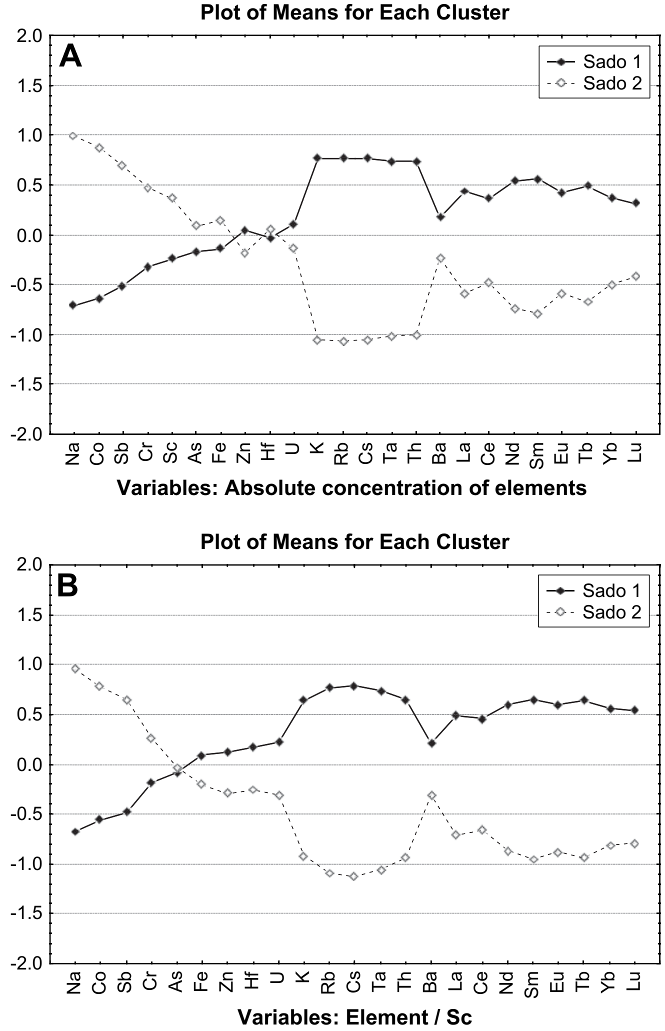

Fig. 3. Plots of means for each variable of the K-means clustering method, using the chemical contents (A) and the Sc-normalized values (B) as variables (HP data from Cabral et al., 2000).

2. Materials and methods

Amphorae sherds (75 samples) of different typologies (Dressel 14, Dressel 14b, Dressel 30, Almagro 50, Almagro 51c, Almagro 51a–b, Keay LXXVIII) from five archaeological sites of the Lower Sado with evidence of ceramics production were selected for this study, namely from Barrosinha (BA), Bugio (BU), Zambujalinho (ZA), Xarrouqueira (XA) and Vale da Cepa (VC). The location of these archaeological sites is shown in Fig. 1, as well as other sites, namely the Herdade do Pinheiro (HP) production centre and the Tróia industrial centre of fish preserves.

Chemical characterization was performed by instrumental neutron activation analysis (INAA) and mineralogical composition of selected samples was determined by X-ray diffraction (XRD). To obtain powder samples for analysis of ceramics, the inner and outer surfaces of the sherds were scraped using a drill burr made of tungsten carbide. The scraped specimens were subsequently brushed clean, washed, boiled for 30 min in deionized water, and then dried for several hours at 30 °C. After drying, a planetary agate

Table 3

F values (variance) of each variable resulting from K-means clustering method using the absolute contents and the Sc-normalized values (HP data from Cabral et al., 2000).

	$F_{\text{Absolute values}}$	$F_{\text{Norm. Sc}}$
Na	**345**	**256**
K	**609**	**193**
Fe	3.01	3.75
Sc	13.6	–
Cr	25.7	6.66
Co	**180**	**108**
Zn	2.94	10.9
As	4.70	0.248
Rb	**632**	**719**
Sb	76.8	71.8
Cs	**591**	**1278**
Ba	5.79	9.54
La	48.4	72.7
Ce	29.1	59.6
Nd	88.5	**149**
Sm	**107**	**219**
Eu	46.7	**153**
Tb	67.5	**204**
Yb	31.6	**117**
Lu	21.1	**100**
Hf	0.305	6.41
Ta	**419**	**469**
Th	**412**	**219**
U	2.31	9.94

mortar for small samples was used to crush and homogenize ~2 g of pottery sample into a fine powder (<53 μm). Aliquots of ~1 g of powder of each sample were then dried in an oven at 110 °C for 24 h and stored in a desiccator until being weighed for INAA. Two multi-element reference materials were used, namely GSS-4 and GSS-5 from the Institute of Geophysical and Geochemical Prospecting (IGGE), to perform chemical analysis by INAA (comparative method). The reference values were taken from data tabulated by Govindaraju (1994). All powdered samples (ceramics and standards) were prepared by weighing 200–300 mg of powder into cleaned high-density polyethylene vials. Long irradiations (7 h) were performed in batches of 20 unknowns along with four standards in the core grid of the Portuguese Research Reactor at a thermal flux of $3.346 \times 10^{12}\ \text{cm}^{-2}\,\text{s}^{-1}$; $\phi_{\text{epi}}/\phi_{\text{th}} = 1.4\%$; $\phi_{\text{th}}/\phi_{\text{fast}} = 12.1$. The bundles were rotated continuously during irradiation to ensure that all samples received the same exposure to neutrons. Furthermore, Fe flux monitors were placed in appropriate plastic containers for irradiation together with the samples and the standards for neutron flux variation corrections.

Two γ-ray spectrometers were used: (1) one consisting of a 150 cm^3 coaxial Ge detector connected through a Canberra 2020 amplifier to an Accuspec B (Canberra) multichannel analyzer. This system has a FWHM of 1.9 keV at 1.33 MeV; and (2) the other consisting of a low energy photon detector (LEPD) connected through a Canberra 2020 amplifier to an Accuspec B (Canberra)

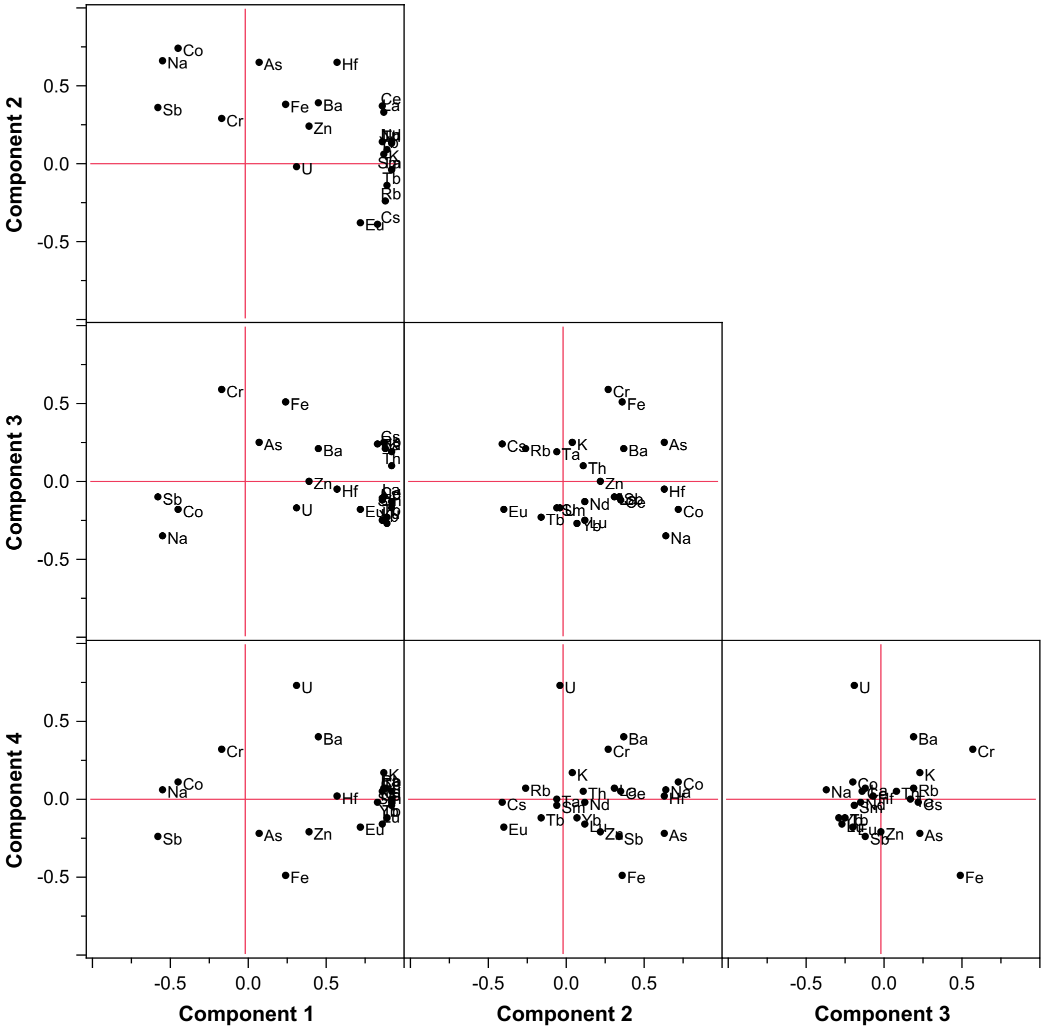

Fig. 4. Bivariate plots of principal components of the first four factors with projections of variables (HP data from Cabral et al., 2000).

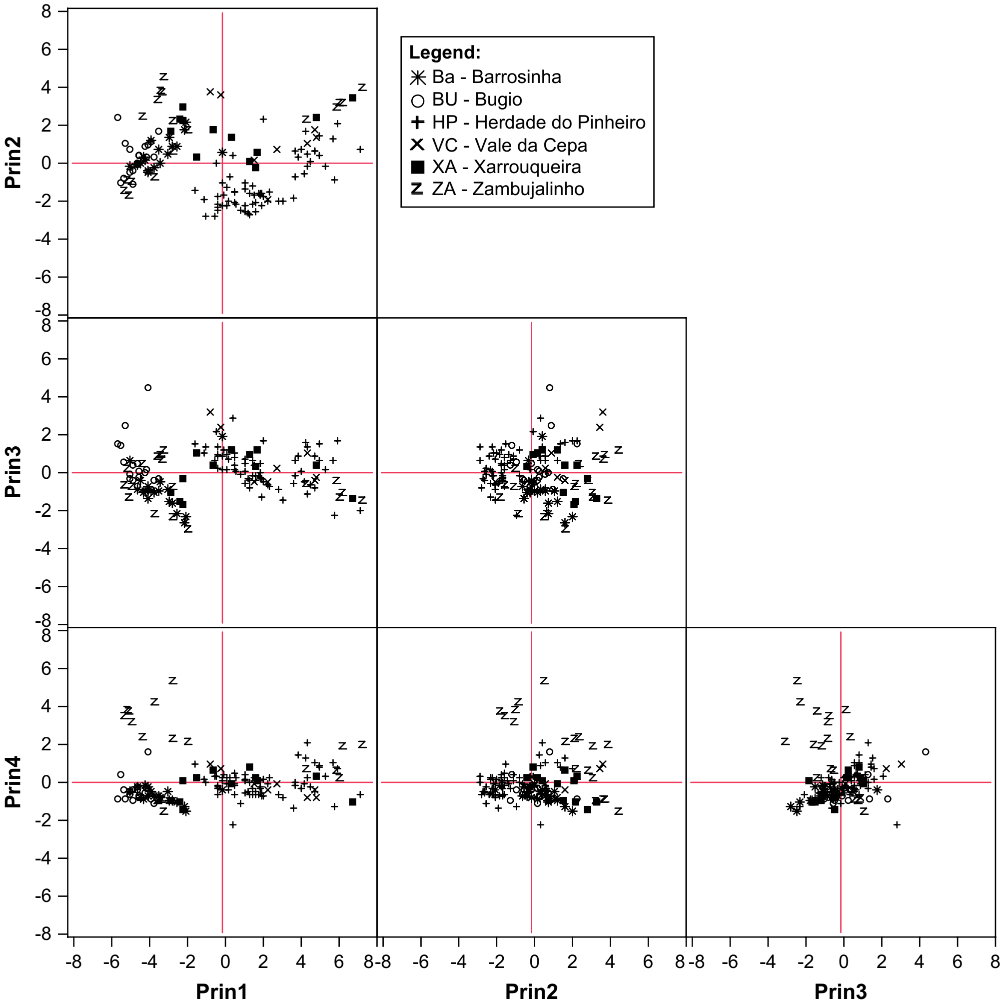

Fig. 5. Bivariate plots of principal components of the first four factors with projections of cases (HP data from Cabral et al., 2000).

multichannel analyzer. This system has a FWHM of 300 eV at 5.9 keV and of 550 eV at 122 keV.

Different cooling times were selected to determine radionuclides with different half-lives. These cooling times and energies of γ-rays, as well as the type of detector used are given in Table 1. Na, K, Fe, Sc, Cr, Co, Zn, As, Rb, Cs, Ba, La, Ce, Nd, Sm, Eu, Tb, Yb, Lu, Hf, Ta, Th and U abundances were obtained. Relative precision and accuracy are, in general, within 5%, and occasionally within 10%.

The spectra were processed by using the programs GELI and DAISY, adapted respectively from GELIAN and OLIVE programs (Op De Beek, 1972, 1974). Two pairs of standards were used in order to obtain an internal calibration. Corrections were made for the interference from uranium fission products in the determination of barium and rare earths (Martinho et al., 1991; Gouveia et al., 1987). More details of the method can be found in Gouveia and Prudêncio (2000).

Multivariate statistical methods were employed by using the Statistica program (StatSoft, 2003) and JMP7 program (SAS). The joining-tree and K-means clustering methods (hierarchical and non-hierarchical, respectively), principal component analysis (PCA) and discriminant analysis using the absolute concentration of the chemical elements as variables, as well as the normalized contents relative to the Sc content ([X]sample/[Sc]sample), were employed. This normalization of the chemical contents relative to Sc (conservative element) was performed in order to compensate for grain size and mineralogy effects on trace element concentrations,

Table 4
Projection of the variables (Sc-normalized values) in the first four principal components (HP data from Cabral et al., 2000).

	F1	F2	F3	F4
Na	**−0.53**	**0.66**	−0.35	0.06
K	**0.89**	0.06	0.25	0.17
Fe	0.26	0.38	**0.51**	−0.49
Cr	−0.15	0.29	**0.59**	0.32
Co	−0.43	**0.74**	−0.18	0.11
Zn	0.41	0.24	0.03	−0.21
As	0.09	**0.65**	0.25	−0.22
Rb	**0.90**	−0.24	0.21	0.07
Sb	**−0.56**	0.36	−0.10	−0.24
Cs	**0.85**	−0.39	0.24	−0.02
Ba	0.47	0.39	−0.21	0.40
La	**0.89**	0.33	−0.10	0.07
Ce	**0.88**	0.37	−0.12	0.05
Nd	**0.95**	0.14	−0.13	−0.02
Sm	**0.94**	−0.04	−0.17	−0.04
Eu	**0.74**	−0.38	−0.18	−0.18
Tb	**0.91**	−0.14	−0.23	−0.12
Yb	**0.91**	0.09	−0.27	−0.12
Lu	**0.88**	0.14	−0.25	−0.16
Hf	**0.59**	**0.65**	−0.05	0.02
Ta	**0.94**	−0.04	0.19	0.00
Th	**0.94**	0.13	0.10	0.05
U	0.33	−0.02	−0.17	**0.73**
Var. (%)	52.9	13.5	6.1	5.8

thus diminishing erroneous interpretation of ceramic characterization and differentiation (Dias and Prudêncio, 2008). Descriptive statistics were also done in order to characterize eventual reference groups created.

Mineralogical composition was determined by XRD, using a Philips X'Pert Pro diffractometer, with a PW 3050/6x goniometer, Cu K_{α} radiation, fixed divergence slit and operating at 45 kV and 40 mA. Powders of non-oriented aggregates were used to obtain the diffraction patterns. Scans were run from 3 to 70° 2θ, using a step size of 0.02° 2θ and a scan step time of 1.20 s. To estimate quantities we measured the diagnostic reflections areas, considering the full width at half maximum (FWHM) of the main minerals and then weighted by empirical factors or calculated parameters (Schultz, 1964; Biscaye, 1965; Martín-Pozas, 1968). Diagnostic peaks used and the corresponding reflection powers (in parenthesis) were the following: quartz – 3.35 Å (2), calcite – 3.04 Å (1), alkali feldspar – 3.25 Å (1), plagioclase – 3.20 Å (1), mica – 10 Å (0.5), phyllosilicates – 4.46 (0.1) and hematite – 2.70 (1.3). Given the uncertainties involved in the semi-quantitative method, the results obtained should only be taken as rough estimates of mineral percentages.

3. Results and discussion

The chemical results obtained for the amphorae sherds from Barrosinha (BA), Bugio (BU), Zambujalinho (ZA), Xarrouqueira (XA) and Vale da Cepa (VC) are given in Table 2.

The chemical composition of the sherds from the five archaeological sites, as well as the results obtained previously by Cabral et al. (2000) for Herdade do Pinheiro (referred as HP in the present work), was used for a multivariate statistical analysis.

Exploratory methods (joining-tree clustering, K-means and PCA) were employed in order to distinguish eventual groups based on the chemical contents as well as using Sc-normalized values as variables.

The joining-tree clustering using the Euclidean distance as the similarity coefficient and Ward's amalgamation method applied to the chemical contents revealed the existence of three outliers, one from Xarrouqueira (XA-4) mainly due to higher Zn and particularly As contents, one from Bugio (BU-17) mainly due to high Ba concentration (see Table 2) and one from Herdade do Pinheiro (HP-61) due to slightly higher rare earth elements (REE) contents.

The joining-tree clustering using the Euclidean distance showed that after normalization, only the sample from Xarrouqueira (XA-4) remains an outlier. In this way only this sample was not considered in the following statistical treatments. The dendogram resulting from the joining-tree clustering using the Pearson correlation coefficient and Ward's method points to the existence of two main groups, named Sado 1 and Sado 2 (Fig. 2). Sado 1 comprises: BA – 1 sample out of 18 sherds (1/18), XA – 8/13, VC – 6/8, BU – 1/18, ZA – 5/18, and HP – all. Sado 2 comprises all samples from BU and BA (except one) and the remaining samples from ZA, XA and VC. A similar result is obtained by using the K-means method ($k = 2$)

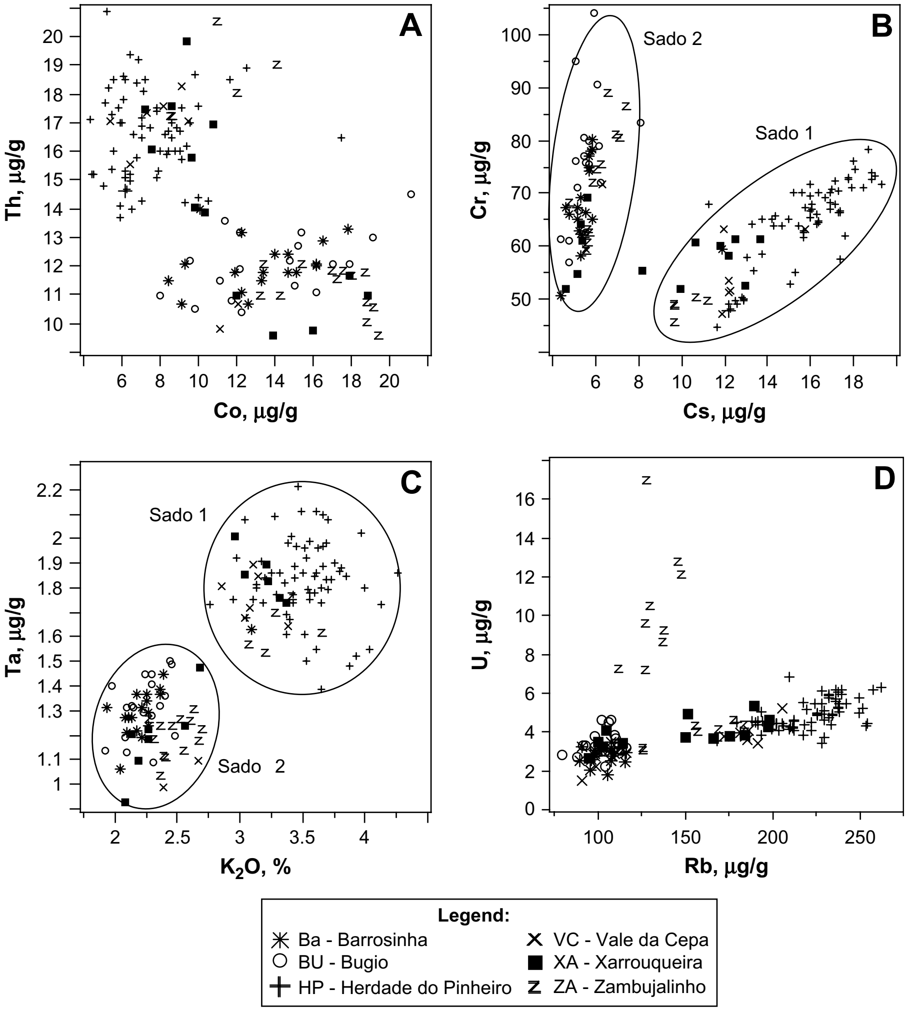

Fig. 6. Bivariate plots of: Co vs. Th (A); Cs vs. Cr (B); K_2O vs. Ta (C); and Rb vs. U (D) (HP data from Cabral et al., 2000).

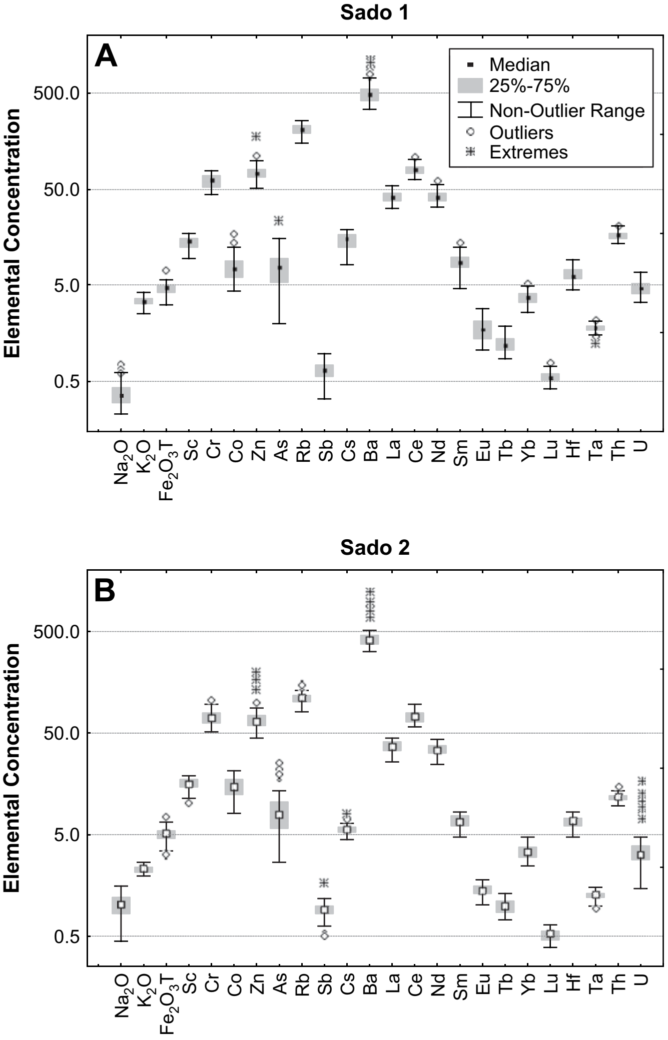

Fig. 7. Box plots for each chemical element studied of Sado 1 (A) and Sado 2 (B) (HP data from Cabral et al., 2000).

using both chemical contents and Sc-normalized values as variables. The contribution of each variable for the differentiation of the two groups is clearly shown in the plots of means for each cluster (Fig. 3). As can be seen in Table 3, where the magnitude of the *F* values (higher for the variables whose inter-cluster variance is higher than the intra-cluster variance) is presented, the two clusters are best differentiated when Sc-normalized data are used. The variables that contribute more significantly to the clusters differentiation ($F \geq 100$) are as following: Cs > Rb > Ta > Na > Sm = Th > Tb > K > Eu > Nd > Yb > Co > Lu.

The principal component analysis (PCA) was applied to the Sc-normalized contents, and the plots of the several projections of variables and samples on the F1–F4 factors (78.3% of the total variance) are shown in Figs. 4 and 5. The factor coordinates of the variables are given in Table 4. The results agree with those obtained by the two clustering methods employed. The variables that contribute more to the first principal component (F1: 52.9% variance) are Ta, Th, REE, Rb, K and Cs. For the second component (F2: 13.5% variance) significant contributions are given by Co, Na, As and Hf. This analysis also showed a significant variation of uranium, particularly in Zambujalinho sherds within the Sado 2 group, shown by the F4 (see Figs. 4 and 5, and Table 4).

The results obtained emphasize the geochemical differences found in the composition of the ceramics paste of the two groups, which appear to be correlated with the nature of the raw materials used. The Sado 1 group presents in general lower contents of Na, Co and Sb and higher contents of other elements studied, particularly Cs, Rb, Ta, Th, and K, as well as REE when compared to Sado 2.

Bivariate plots of chemical elements with different geochemical behaviours that better distinguish both groups, as also revealed by the multivariate statistical analysis, are shown in Fig. 6. Among these, plots of Cs vs. Cr and K_2O vs. Ta can be used to distinguish the Sado 1 and Sado 2 groups. The Rb vs. U plot clearly shows the high contents of U found in some sherds of Zambujalinho from Sado 2 group.

Box plots for each chemical element of both groups are shown in Fig. 7. It should be noted that outliers and extreme values are found particularly in the Sado 2 group for Zn, As, Ba, and U. In the Sado 1 group extreme values are mainly found for Ba. Non-outlier ranges

Table 5
Average concentration values ($\bar{x}$) and corresponding standard deviations (σ), minimum and maximum values for each chemical element (µg/g unless expressed otherwise) for Sado 1 and Sado 2 groups (HP data from Cabral et al., 2000).

	Sado 1					Sado 2				
	n	$\bar{x}$	Min.	Max.	σ	*n*	$\bar{x}$	Min.	Max.	σ
Na_2O, %	83	0.365	0.230	0.620	0.091	54	0.998	0.442	1.54	0.240
K_2O, %	86	3.41	2.55	4.25	0.323	54	2.29	1.92	2.68	0.181
Fe_2O_3T, %	85	4.65	3.14	5.76	0.595	50	5.03	3.38	6.51	0.754
Sc	86	13.9	9.39	17.2	2.06	53	15.6	11.2	19.0	1.85
Cr	86	61.9	44.6	78.3	8.67	53	70.5	50.8	94.8	9.74
Co	84	7.59	4.29	12.4	1.86	54	14.5	7.99	21.1	3.21
Zn	84	74.0	51.2	99.9	10.7	50	64.5	43.9	88.6	10.1
As	85	7.68	1.99	15.6	2.97	48	7.68	2.66	13.4	2.70
Rb	86	212	151	263	26.6	50	107	81.1	131	11.3
Sb	86	0.647	0.328	0.968	0.141	51	0.905	0.614	1.17	0.114
Cs	86	14.6	8.08	19.2	2.63	50	5.45	4.37	6.48	0.495
Ba	75	477	337	716	84.8	47	401	311	506	49.8
La	86	41.4	31.9	53.7	4.56	54	36.8	25.7	48.2	4.31
Ce	85	79.5	63.4	103	8.38	54	72.5	57.4	96.6	8.57
Nd	85	42.1	32.3	55.5	5.54	54	34.4	24.5	43.0	4.33
Sm	85	8.76	4.56	12.5	1.50	54	6.72	4.70	8.19	0.928
Eu	86	1.78	1.07	2.88	0.458	54	1.40	1.01	1.76	0.182
Tb	86	1.24	0.852	1.85	0.225	54	0.996	0.715	1.32	0.142
Yb	85	3.69	2.63	4.90	0.508	54	3.32	2.42	4.64	0.441
Lu	85	0.555	0.411	0.714	0.074	54	0.513	0.379	0.639	0.061
Hf	86	6.54	4.54	9.22	1.15	54	6.64	4.72	8.38	0.827
Ta	82	1.81	1.48	2.11	0.149	53	1.26	0.983	1.50	0.118
Th	86	16.7	13.7	20.9	1.60	53	11.6	9.51	13.6	0.968
U	86	4.73	3.36	6.87	0.781	45	3.06	1.47	4.63	0.649

are larger for As, Co, Na and Sb for both ceramics groups and U for Sado 2.

Taking into account the results obtained by the several methods employed, statistical parameters of chemical concentrations for the two clusters to be used as reference groups for the Sado estuary are given in Table 5.

The mineralogical composition of selected sherds belonging to the two groups is given in Table 6. Quartz, alkali feldspars, plagioclase, phyllosilicates, micas, calcite and hematite could be identified by XRD. Samples of the Sado 2 group are clearly differentiated by the presence of plagioclase, besides alkali feldspars. Micas and phyllosilicates' proportions do not differentiate the two groups. However, these mineral phases should not be considered for raw materials inferring since their structure may have been destroyed during firing, and its identification and semi-quantification by XRD mostly reflect differences in the temperature raised in the kilns.

The presence of plagioclase associated with the higher contents of sodium in the sherds of the Sado 2 group point to the use of raw materials with a more basic composition when compared to the Sado 1 group. These characteristics of the Sado 2 group, as well as the high contents of Co, Sb, As and Zn found in some samples, point to a diverse composition of the source area of the sediments used as raw materials. It should be noted that sample XA-4 becomes an outlier due to high contents of Zn and particularly As, even after Sc-normalizing. However, if one substitutes the concentrations of As and Zn for the mean values for As and Zn and redoes the statistical analysis, this sample is included in the Sado 2 group. Thus, the sediments used to produce amphorae of Sado 2 group appear to have a significant contribution of basic rocks such as gabbros and diorites, and also of mineralized areas (Iberian Pyrite Belt) occurring in the Sado basin. In this way sherds found in reception centres with a composition similar to the one found for Sado 2 group with high contents of As, Zn, Co and Sb may not be dismissed as not having a Sado provenance, since occasional grains containing primary or secondary minerals of sulfides may occur in the ceramics paste. The Sado 1 group is clearly differentiated by higher contents of K, Rb and Cs, reflecting the higher proportion of alkali feldspars relative to plagioclase. The higher contents of Ta, together with the high concentrations of the above referred elements, may be also an indicator of a higher proportion of micas in the raw materials used, when compared to the clays employed to manufacture amphorae of Sado 2 group. High contents of Ba in some samples of both groups may be partially related with the presence of barite.

The high contents of uranium found in some sherds of Zambujalinho may be explained by the mixture of raw materials with brackish clays, where high contents of U may occur associated with organic matter in this estuary environment. It should be noted that these sherds also present other 'anomalous' values such as low iron contents relative to Sc. The results obtained for this archaeological site, located in the upper tributary of the Sado River (see Fig. 1), suggest a 'local anomaly' that needs further confirmation with a larger number of sherds analyzed.

Table 6
Estimated mineralogical composition for selected sherds of Sado 1 and Sado 2 groups.

Sample	Quartz	Phyllosilicates	Alkali-feldspar	Plagioclase	Calcite	Mica	Hematite
Sado 1							
BA-14	55	36	5	–	tr	3	–
VC-4	63	21	10	–	tr	5	–
VC-17	89	–	7	–	–	–	4
XA-20	96	–	3	–	–	–	tr
ZA-17	92	–	8	–	tr	–	–
Sado 2							
BA-18	63	21	5	7	–	3	tr
BA-19	95	–	4	tr	–	–	tr
BU-16	49	32	9	4	–	6	–
VC-6	49	31	3	3	8	6	–
VC-12	43	24	4	3	22	4	–

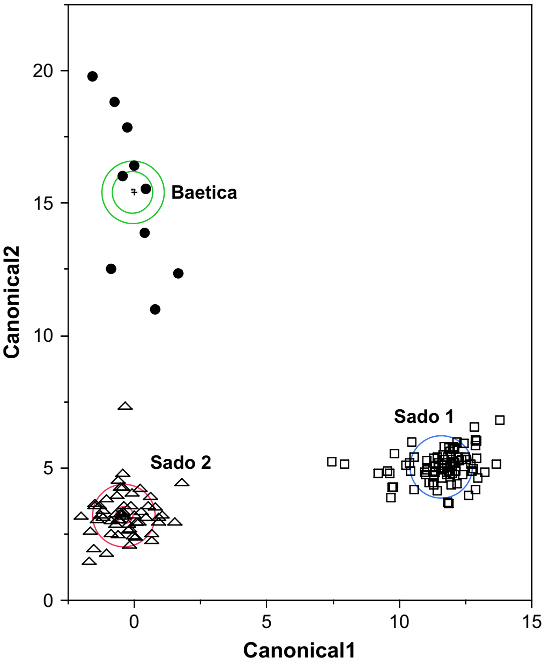

Fig. 8. Plots of canonical values (1 and 2) of the discriminant analysis (HP data from Cabral et al., 2000).

Discriminant analysis using the chemical composition (Sc-normalized values) applied to the amphorae from Sado River and Guadalquivir River (Villa Nueva, Cádiz) allows to distinguish the Baetica amphorae from the two compositional groups of Sado, as shown in Fig. 8. The identification of multiple groups using discriminant analysis followed by a canonical correlation analysis made it possible to visualize how those functions discriminate between the two productions of the Sado estuary and amphorae produced in Baetica. Among the elements studied Cr contents normalized to Sc better differentiate Cádiz (Villa Nueva) from Sado productions (see Tables 7 and 8).

Amphorae found in underwater environments of both coasts of the Straits of Gibraltar with high Cr contents were already referred by de la Villa et al. (2003) as coming from Cádiz, and used to distinguish from Ceuta productions. However, the Cr contents obtained by those authors (<49 μg/g) are lower than the concentrations obtained by INAA for amphorae from Villa Nueva, Cádiz (84–188 μg/g). It should be noted that a very small number of amphorae sherds (10) of only one archaeological site of the Guadalquivir estuary were already analyzed by INAA at ITN (Dias et al., 2003) and used in this work for comparative purposes. In order to obtain a better characterization of amphorae produced in the margins of the Guadalquivir River and their differentiation from other amphorae-producing areas, particularly the Lower Sado due to the similarity of the lithological composition of both river basins,

Table 7
Means of canonical variables (Sc-normalized values) resulting from the discriminant analysis (HP data from Cabral et al., 2000).

	Root 1	Root 2
Sado 1	5.13	−0.03
Sado 2	−6.83	−1.84
Baetica	−6.53	10.4

Table 8
Standardized coefficients for canonical variables (Sc-normalized values) resulting from the discriminant analysis (HP data from Cabral et al., 2000).

	Root 1	Root 2
Na	−0.21	**0.56**
K	0.10	−0.04
Fe	−0.25	0.19
Cr	−0.12	**0.95**
Co	0.20	−0.05
Zn	0.14	0.24
As	−0.07	−0.24
Rb	0.06	−0.41
Sb	−0.12	−0.45
Cs	**0.71**	0.42
Ba	−0.22	−0.25
La	**−0.94**	−0.19
Ce	**−1.46**	**−0.69**
Nd	**1.87**	**0.79**
Sm	−0.37	0.01
Eu	−0.34	−0.31
Tb	**0.67**	**0.62**
Yb	**−0.71**	**−0.60**
Lu	0.03	−0.22
Hf	**−1.02**	−0.01
Ta	0.31	0.25
Th	**2.05**	0.30
U	−0.19	0.01
Eigenvalues	35.5	8.55
Cum. Prop.	0.81	1.00

will require a larger data set, including other archaeological sites of the Cádiz Bay.

4. Conclusions

The application of multivariate statistical analysis to chemical data obtained by INAA of Roman amphorae found in several archaeological sites of the Lower Sado which show evidence of ceramic production, led us to establish two main compositional groups: Sado 1 with low contents of Na, Co and Sb and high contents of Cs, Rb, Ta, Th, K and REE, when compared with the second group Sado 2. The geochemical patterns of the two ceramics groups indicate the use of two different types of raw materials, one more felsic (Sado 1) and the other more mafic (Sado 2). The mineralogical composition obtained by XRD agrees with the chemical contents of these two groups. The choice of raw materials appears to be related only with the proximity of the kilns, since no evidence of correlation between the typology and the chemical composition was found.

Chromium contents, particularly after normalizing to Sc, can be used as an indicator to differentiate amphorae produced in the Lower Sado (Lusitania province) from amphorae produced in Villa Nueva, Cádiz, Spain (Baetica province), since higher Cr/Sc values were found in ceramics produced in the archaeological site of the Guadalquivir estuary.

The creation of reference groups of amphorae from the Lower Sado (Sado 1 and Sado 2), and the establishment of the respective indicators will allow to differentiate them from ceramic vessels produced in other areas of the Roman Empire, and at the same time to reconstruct short and long-distance supplying and consumption routes originated in Lusitania.

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