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► To cite this version:

Valérie V. Thirion-Merle, Gisela Thierrin-Michael, Armand Desbat. Archaeometrical study of Khmer stoneware from the Angkorian period: some results of the Cerangkor Project. 14th International Conference of the European Association of Southeast Asian Archaeologists (EurASEAA 14), University College Dublin, Sep 2012, Dublin, Ireland. hal-01394076

HAL Id: hal-01394076

<https://hal.science/hal-01394076>

Submitted on 8 Nov 2016

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Archaeometrical study of Khmer stoneware from the Angkorian period: some results of the Cerangkor Project

Valérie Thirion-Merle, Gisela Thierrin-Michael and Armand Desbat

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Abstract

In the area around Angkor, a number of ceramic kilns dating from the 9-15th centuries AD have been discovered since 1995. The technical, typological and compositional characterization of their production has been one of the main goals of the Cerangkor Project (Desbat *et al.* 2008, 2009, 2010, 2011 and 2012).

Since 2008, more than 300 samples of Khmer stoneware produced in the Angkor region have been analyzed chemically by XRF-WDS, and forty-three of these have also seen petrographic analysis. The ceramics comprise green-glazed 'Kulen type' stoneware and non-glazed stoneware from five kiln sites (Anlong Thom, Sor Sei, Tani, Khnar Po and Bankaong), as well as brown-glazed stoneware from the Anlong Thom site. The data indicate that some workshops used quite similar raw materials for the same types of ceramics, suggesting the exploitation of the same geological formations for the whole region. Differentiation of the compositional groups characterizing each workshop proved, therefore, to be fairly difficult, but small differences allow us to distinguish between some groups of workshops.

Introduction

The archaeometrical study of Khmer stoneware presented here has been carried out since 2008 as part of the Cerangkor Project. This project, created in 2008, is a collaborative research program between the CNRS (National Center for Scientific Research of France), the EFEO Center in Siem Reap (French School of Asian Studies) and the APSARA Authority (Authority for the Protection and Management of Angkor and the Region of Siem Reap) with funding of the Ministry of Foreign Affairs of France (Desbat *et al.* 2008, 2009, 2010, 2011, 2012). It was developed to investigate the ceramics produced at the kiln sites in the region of the ancient Khmer capital of Angkor, from the period between the 9-15th centuries AD. In this paper, we concentrate on the results concerning the production of the kiln sites Anlong Thom and Sor Sei, situated respectively on and at the foot of Phnom Kulen, as well as Khnar Po, Tani and Bankaong, between Phnom Kulen and the lake of Tonlé Sap (Fig. 1). (Aoyagi. *et*

al. 2000; Aoyagi and Sasaki 2006; Tabata and Chhay Visoth 2007; Miksic *et al.* 2008; Ea Darith 2010). All these sites are situated in the Siem Reap Province.

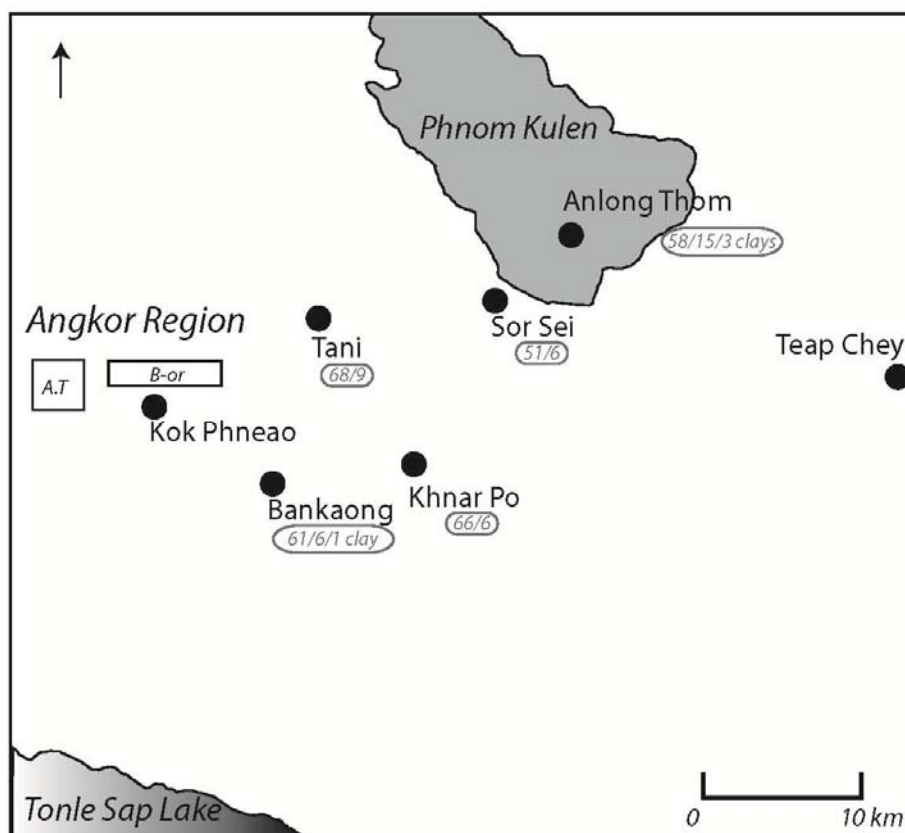


Figure 1 : Geographical situation of the kiln sites; number of analyzed samples in brackets (chemical analyses/petrographical analyses); AT Angkor Thom, B-or = eastern Baray

Site context and sample collection

The pottery samples discussed here were collected on these kiln sites during our own field prospectations. Clay and sand samples for comparison were taken either directly at the kiln sites or from locations situated within perimeters under 5 km around the kiln sites. All of the kiln sites studied are characterized by the presence of huge quantities of sherds, distributed in several manners at the sites: in Anlong Thom, the sherds are dispersed in the forest as well as concentrated in the partially-excavated kiln structures due to the villagers's looting (Desbat *et al.* 2008); in Tani, Sor Sei and Khnar Po the ceramics were concentrated around the kiln remains, which appear as mounds in the landscape (Desbat *et al.* 2008, 2009 and 2010), while the dispersal zone of sherds around the kilns is larger at the site of Bankaong (Desbat *et al.* 2008). Some kilns at each site were excavated prior to this project (Aoyagi. *et al.* 2000; Aoyagi and Sasaki 2006; Tabata and Chhay Visoth 2007; Miksic *et al.* 2008; Ea Darith 2010). At Khnar Po, several kilns were destroyed and cut at the level of the firing chamber during road works and field remodeling, and are now plainly visible (Desbat *et al.* 2009). At most kiln sites several types of wares were produced: unglazed stoneware, green-

glazed stoneware and earthenware. In addition, brown glazed stoneware was produced at Anlong Thom, while unglazed ware was rarely found there (Desbat *et al.* 2008).

Goals and methods

The main goal of this research was the creation of a reference database for future provenance studies. This meant characterizing kiln production chemically and petrographically, as well as attempting to differentiate the workshops. The study also aimed to increase our technological information concerning raw materials and ceramic paste preparation, and to answer the following questions: what kind of clay(s) was/were used; is it possible to identify the location of the clay resources; and how was clay prepared (washing/levigating, mixing with sand or of several clays, differences between wares, etc.)?

Two analytical methods were combined: chemical and petrographical analyses. The main analytical method applied was chemical analyses by Wavelength Dispersive X-Ray Fluorescence Spectrometry (WD-XRF), according to the standard procedures of the Archaeometry and Archaeology Laboratory at CNRS–University of Lyon. This procedure supplies the bulk chemical composition of the ceramic (matrix and sandy fraction) and consequently of the material used for its manufacturing (Thirion-Merle 2014, Waksman 2014). XXX). Measurement is carried out on glass tablets of homogenous composition containing 800 mg of powdered sample. For each sample, twenty-four components were determined. Several uni-, bi- and multivariate statistical data treatments have been applied to interpret the chemical results. Among the multivariate methods used, here we present results mainly from cluster analysis, a statistical treatment using Euclidian distances, calculated in this case on sixteen chemical components: magnesium oxide (MgO), alumina (Al₂O₃), silica (SiO₂), potassium oxide (K₂O), calcium oxide (CaO), titanium oxide (TiO₂), manganese oxide (MnO), iron oxide (Fe₂O₃), vanadium (V), chrome (Cr), nickel (Ni), rubidium (Rb), strontium (Sr), zirconium (Zr), baryum (Ba), and cerium (Ce). The dendrogram produced (see for exemple figure 5) represents the relative distances between samples, where the most similar samples are linked at the smallest distances. (see Picon 1984; Baxter 1994)

The petrographical analyses on thin sections under the polarising microscope were applied to a reduced number of samples. They were chosen because they are representative of the different chemical compositional groups of each kiln site. This method shows the nature, frequency and shape of the inclusions contained in the ceramics as well as the texture of the matrix. A slice of about four mm is cut through the sherd at right angles to the rim (or turning direction), and the wall of this is used for sample preparation. This slice is then mounted on a glass slide and ground down to a standard thickness of 30 μm.

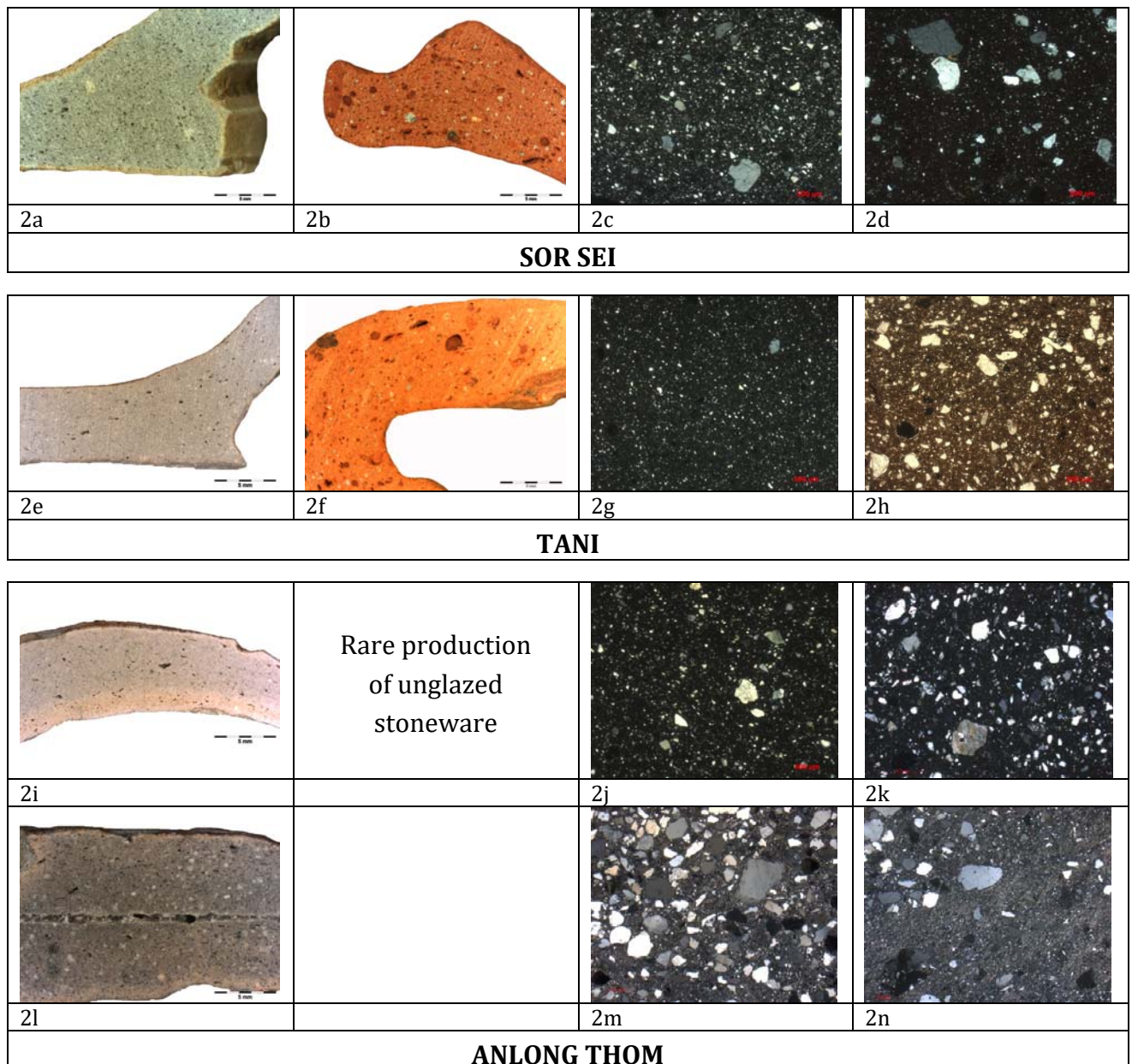
Over 300 samples from the kiln sites were analyzed chemically, as well as clay samples, modern bricks and ceramics. Forty-three of the chemically-analyzed sherds, as well as some clay and sediment samples were studied petrographically under the microscope. The number of analyses per kiln site is shown in Figure 1.

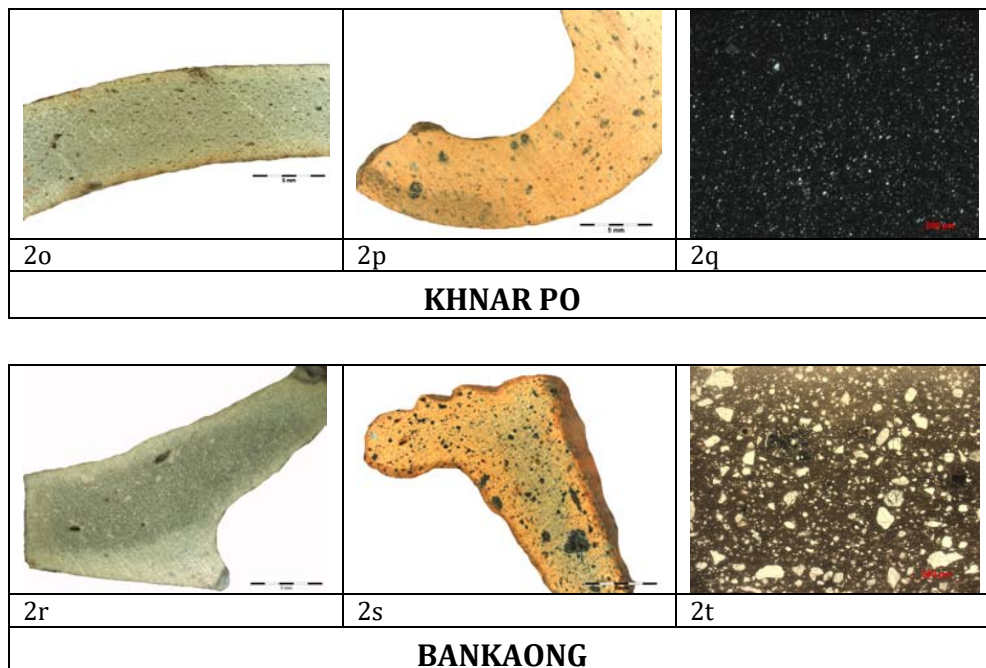
Results

Comparison between wares

To the naked eye, the green-glazed and unglazed stoneware and earthenware present quite different fabrics (Fig. 2). Fabrics of the green-glazed ware are fine-grained and generally light-colored, while the unglazed ceramics as well as the earthenware show coarser, orange to red fabrics with ferruginous nodules. From visual inspection alone, it cannot be determined whether the color differences are due to different iron content or to different firing atmospheres, or to both. In addition it was not clear whether the granulometric differences noted indicate quite different clay sources or different preparations of basically the same clay.

Figure 2 : Fabrics of the glazed and unglazed stoneware, and details of clay sample KHM351 (2m, 2n): macroscopic and microscopic views (XPL, except 2h and 2t; same magnification for all). Green-glazed: 2a/2c, 2e/2g, 2i/2j, 2o/2q, 2r; biscuit: 2k; brown-glazed: 2l; unglazed: 2b/2d, 2f/2h, 2p, 2s/2t.





Under the microscope, both granulometric differences related to inclusions, and differences in the clay matrices were apparent. In the fine-grained fabric noted above, the matrix contains many very fine inclusions which are not visible to the naked eye, while the matrix in the coarser pottery is almost devoid of such fine grains (Fig. 2). This indicates clearly that the two types of wares are made from different clays, as this difference can result neither from levigating the coarser clay used for unglazed stoneware, nor from adding sand to the finer clay used for the green-glazed ware. Levigating or washing a coarse clay will diminish or eliminate the coarse fraction, but not add fine inclusions; adding sand to a clay containing many fine inclusions will result in a coarse paste also rich in fine inclusions (biblio à rajouter par Gisela). The nature of the inclusions, however, almost exclusively grains of quartz, is the same in all analyzed samples.

Chemically, green-glazed and unglazed wares from the same workshop form two groups clearly differentiated by several of the analyzed parameters, as demonstrated in Figure 3 by an example of two parameters. While both wares possess typical stoneware compositions characterized by the high silica (SiO_2) and alumina (Al_2O_3) content usual for clays rich in kaolinite (the chemical formula of which is $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$), the unglazed ware always shows higher contents of iron (Fe_2O_3) (plus some of its correlated elements), as well as potassium (K_2O) and correlated rubidium (Rb). (See table 1). Because of these differences, the comparison between workshops is discussed below separately for each type of ware.

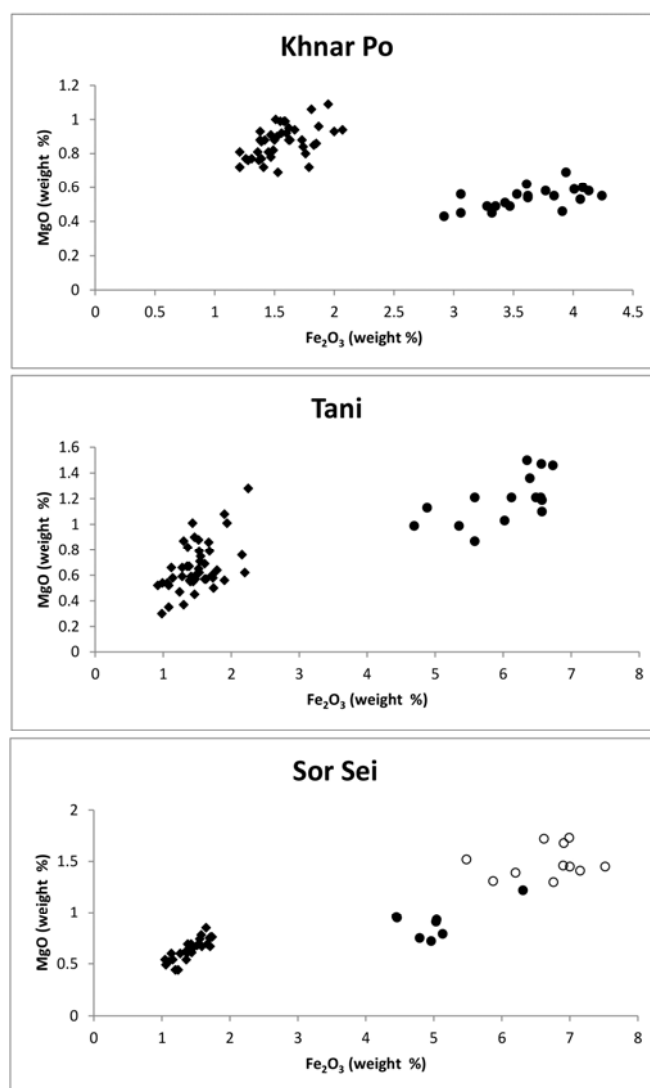


Figure 3 : Binary plots of MgO/Fe₂O₃ with all analyzed samples of green-glazed (diamonds) and unglazed stoneware/earthenware (circles) for each workshop.

Green-glazed stoneware

The green-glazed stoneware of the so-called ‘Kulen type’ was produced at five of the studied kiln sites (Tabata 2005; Ea 2009, 2010). The typological variety seen is presented in Figure 4; urns and boxes with lids of spectacularly-elaborated designs, bottles, and bowls are among the most important forms represented in the sample collection (Desbat 2008, 2009, 2010, 2011, 2012). Chronologically, the earliest vessels come from the workshops of Khnar Po and Tani (Desbat in these proceedings).

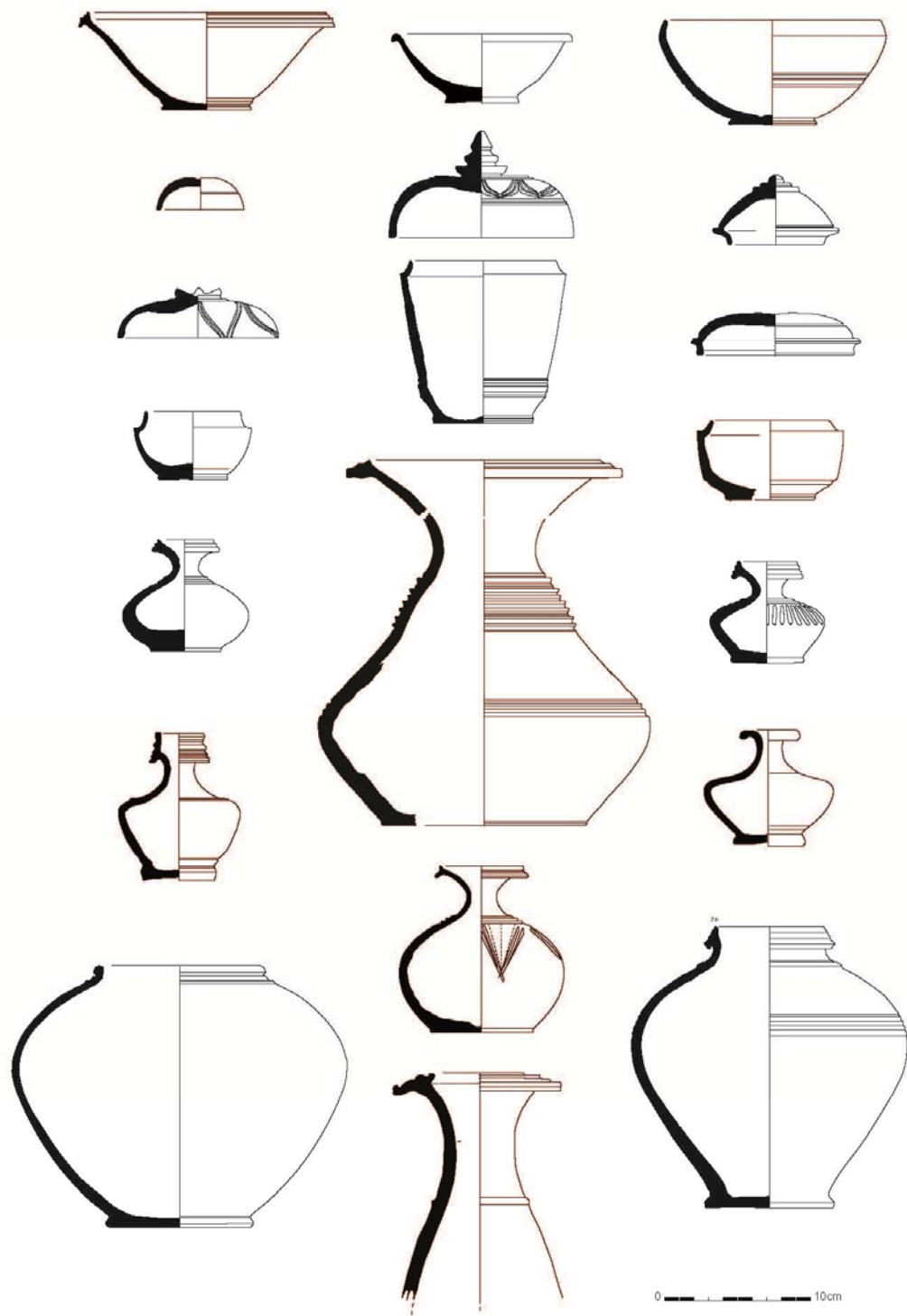


Figure 4 Most important forms of green-glazed stoneware from the different kiln sites.

The chemical composition of this ware is characterized by high silica and alumina contents and very low contents of most other components (table 1). This is typical of ceramics made of quite pure, white-firing kaolinitic clays. These common characteristics indicate that this type of clay was used in all of the five workshops to fabricate green-glazed stoneware. The question was then whether it is possible to further distinguish these ceramic productions by their chemical compositions and/or petrography in spite of the similar raw material.

In order to classify the analyses and to test resemblances, multivariate statistical data treatment was undertaken. The dendrogram of the cluster analysis shows that samples from one workshop are not grouped strictly together (Fig. 5). The analyses separate into two main clusters, the first composed essentially of samples from Khnar Po and Tani, the second of samples from Sor Sei and Anlong Thom. A subgroup from Anlong Thom, chemically distinct from the main group, clusters with the Khnar Po/Tani samples, while a subgroup from Tani is found in the Sor Sei/Anlong Thom cluster. The samples from Bankaong are distributed over the two clusters.

This separation into two main groups is due to small differences in some parameters, principally vanadium, iron, rubidium, silica, zirconium and cerium (Fig. 6). From the cluster analysis it is apparent that the samples from Bankaong do not form a coherent group, which is also demonstrated in Figure 6, where the points representing pieces from that site scatter over the entire variation field occupied by the two ensembles. Although collected at one kiln site, these few samples of green-glazed wares seem to be not of local production at Bankaong, but imported from other workshops.

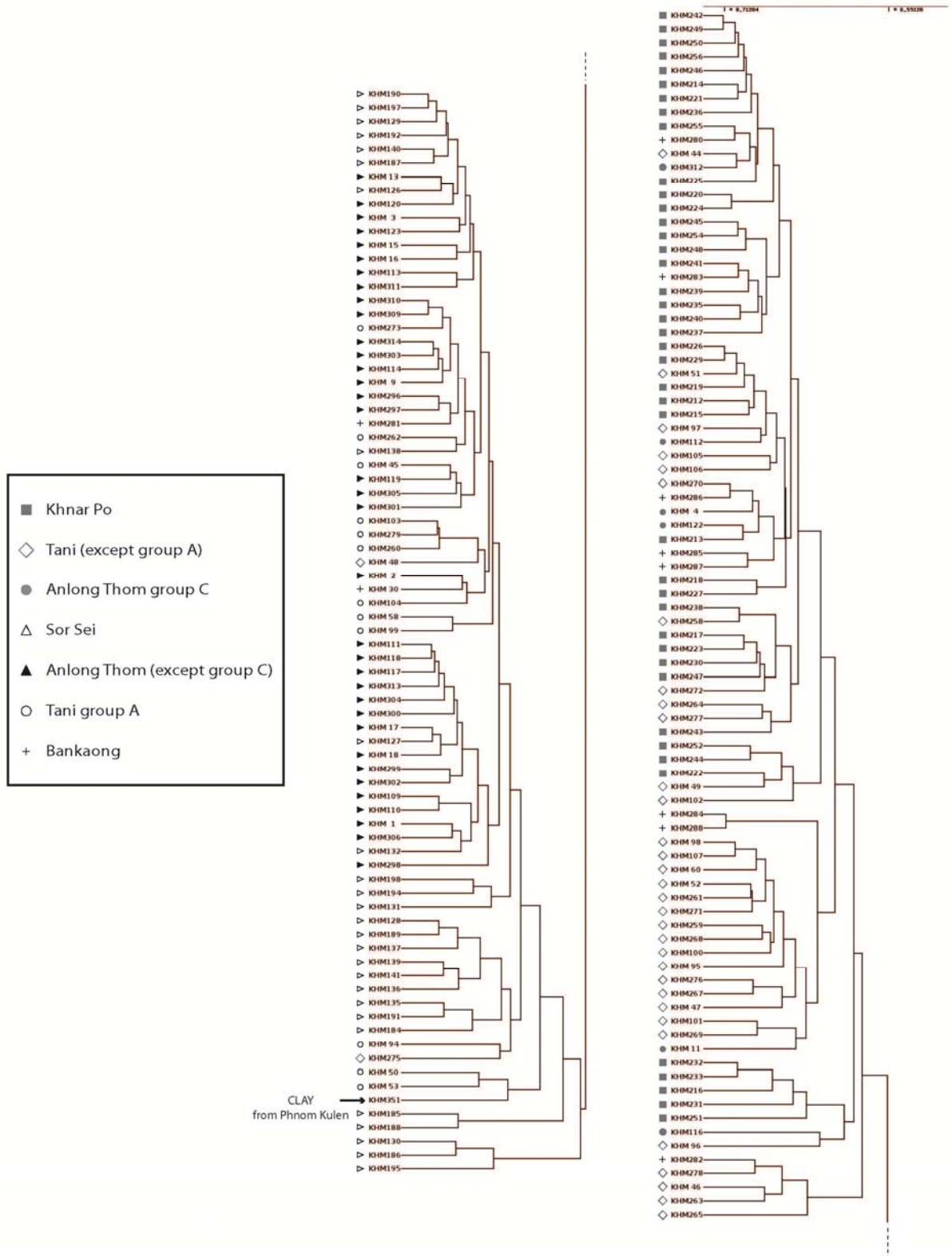


Figure 5 : Dendrogram of cluster analysis of all analyzed green-glazed stoneware from Tani, Bankaong, Anlong Thom, Sor Sei and Khnar Po (calculated on 16 components).

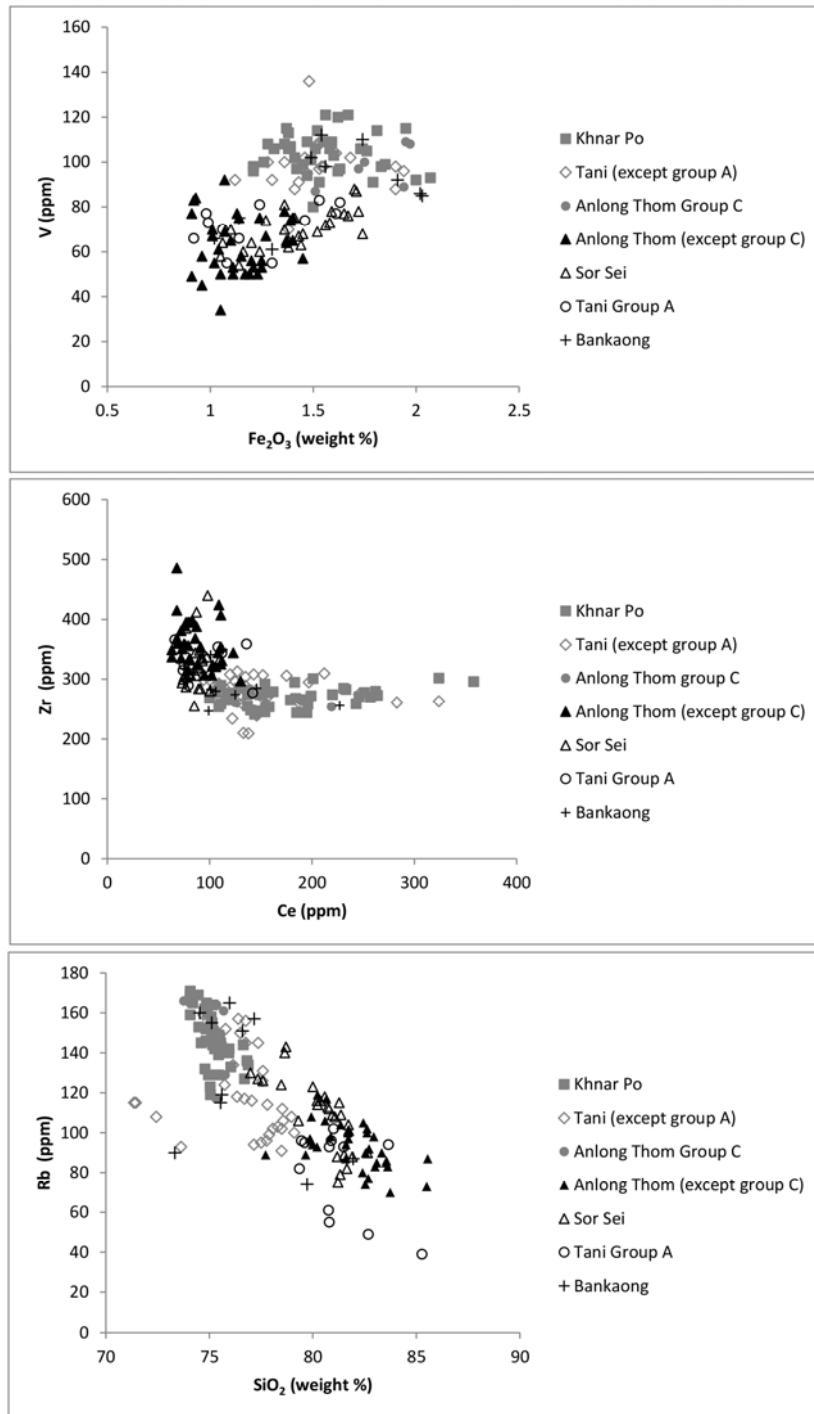


Figure 6 : Selected binary plots with all analyzed samples of green-glazed stoneware.

The ensembles formed on the basis of chemical resemblance can also be distinguished by their granulometry. The fabric of the pieces making up the Anlong Thom/Sor Sei ensemble contains inclusions of various sizes (silt to coarse sand size), while the fabric of those forming the Khnar Po/Tani group mostly shows fine-grained inclusions (silt to fine sand size) and rare larger grains (medium sand size; Fig.2). As mentioned above, the nature of the inclusions, mainly quartz, does in this case not contribute to the differentiation.

In the course of field prospection, clays were collected and analyzed. Among these, one clay source on the Phnom Kulen could be matched chemically to the green-glazed stoneware group from Anlong Thom and Sor Sei. It is marginally integrated into the cluster (Fig. 5) and its granulometry also fits with that group (Fig. 2m, 2n). The details shown in Figure 2 seem to underline the variability in the clay source; it has to be mentioned, however, that the clay sample was not processed prior to thin section preparation (no kneading, wedging or similar treatments). No other clay source for white-firing stoneware, corresponding to our other group, has as yet been discovered.

In summary, the results concerning the stoneware suggest that two groups can be distinguished and defined analytically; each group is composed of most of the representatives of two workshops plus a subgroup from a third (Fig. 7). We also found that the different workshops are not neatly separable into analytically-defined groups, and that very similar kaolinitic, white-firing clays were used at all workshops. Clay for the Anlong Thom/Sor Sei group was well-homogenized prior to pot forming, but was not otherwise processed.

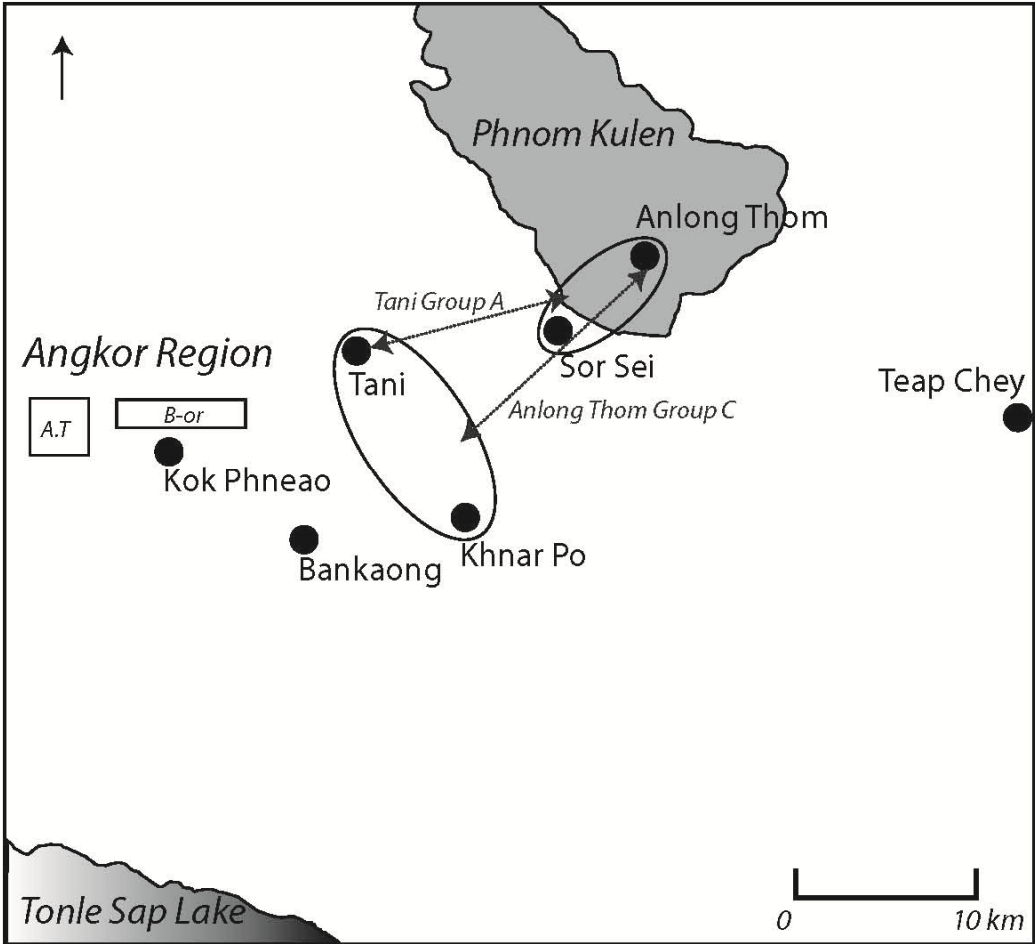


Figure 7 Relationship of green-glazed stoneware production projected onto basemap (ovals); arrows show dissident subgroups.

Geographically, the relationship between the workshops appears as presented in Figure 7. This picture could indicate a geological basis for the compositional differences of the clays used, as both workshops next to Phnom Kulen fit together and may have used the same clay pit, different from the outcrops used by the workshops situated in the plain. However, chronological factors may have played a role as well. This possibility cannot be verified on the basis of our sample collection (surface collection).

Unglazed stoneware and earthenware

The unglazed stoneware and earthenware show similar compositions and are therefore discussed together here (table 1). Analyses on these wares were carried out for four of the workshops: Tani, Sor Sei, Khnar Po and Bankaong. At Anlong Thom this type of ceramic was found only in very small numbers, and therefore has not been analyzed. The analyzed sample collection comprises mainly larger bottles and jars in stoneware, bowls in earthenware (Desbat 2008, 2009, 2010, 2011, 2012).

While products from Bankaong and Khnar Po show light-coloured fabrics (yellow to salmon-coloured), those from Sor Sei and Tani display darker, orange to red colors (Fig. 2). Under the microscope no obvious differences were observed apart from the color, although Sor Sei wares appear to contain the coarsest inclusions from the wares analysed (no petrographic analysis was carried out on ceramics from Khnar Po). Inclusions are almost exclusively quartz, plus different types of ferruginous nodules.

Figure 8

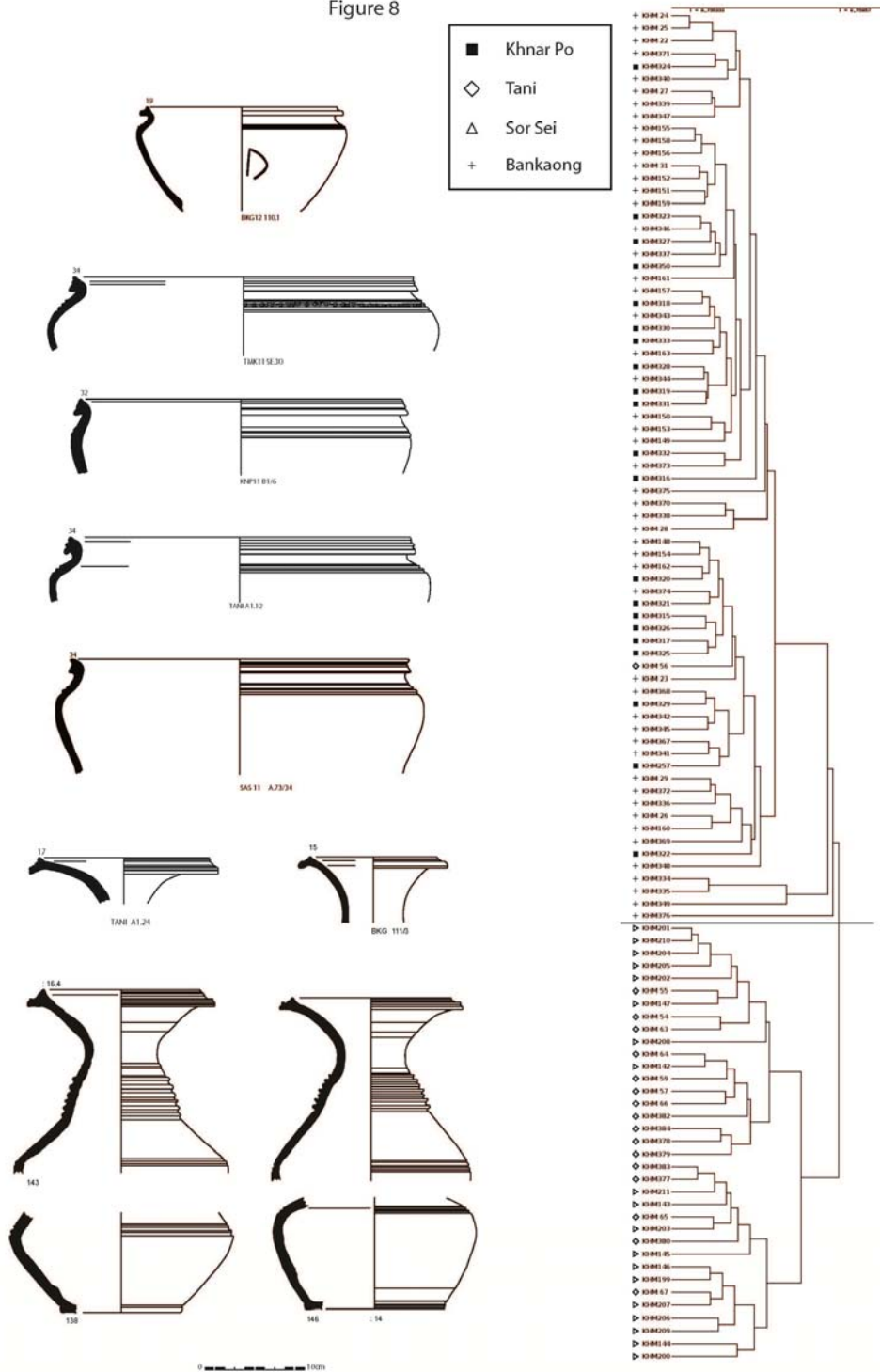


Figure 8 : Dendrogram of the cluster analysis of all analyzed unglazed stoneware and earthenware from Tani, Bankaong, Sor Sei and Khnar Po (calculated on 16 components); typical forms of unglazed stoneware from these kiln sites.

With only one exception, the dendrogram of the cluster analysis reflects this separation into two groups (Fig. 8). Samples from Khnar Po and Bankaong, showing the light fabric, are together in cluster one, and those from Tani and Sor Sei, with the darker fabric, in cluster two.

The separation is due not only to the iron content; other parameters such as potassium oxide, the correlation of alumina and silica, as well as titanium and magnesium oxides contribute as well, as demonstrated by the distribution in the binary plots of Figure 9.

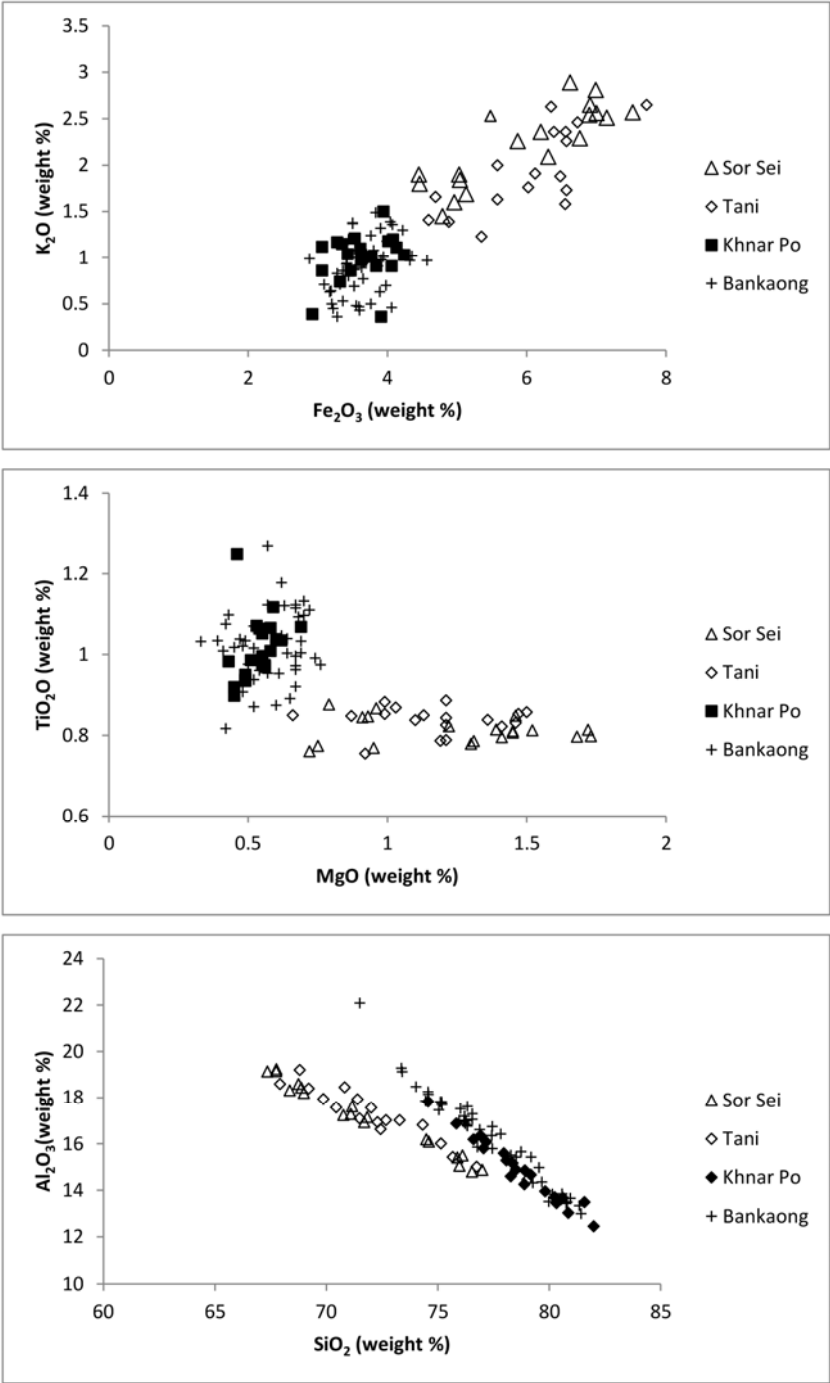


Figure 9 Selected binary plots with all analyzed samples of unglazed stoneware and earthenware.

Projected on the map, the correspondences concerning the unglazed ware mark again two groups, but with different connections compared to the green-glazed ware (Fig.10). Tani and Khnar Po are linked by the use of very similar clays for the green-glazed ware, but do not show this connection concerning unglazed stoneware and earthenware. The relationships between the workshops, based on the use of similar raw materials, appear to be quite different for the unglazed stoneware. As no potential raw materials for these wares have been discovered during field prospection as yet, no explanation for these facts are proposed here.

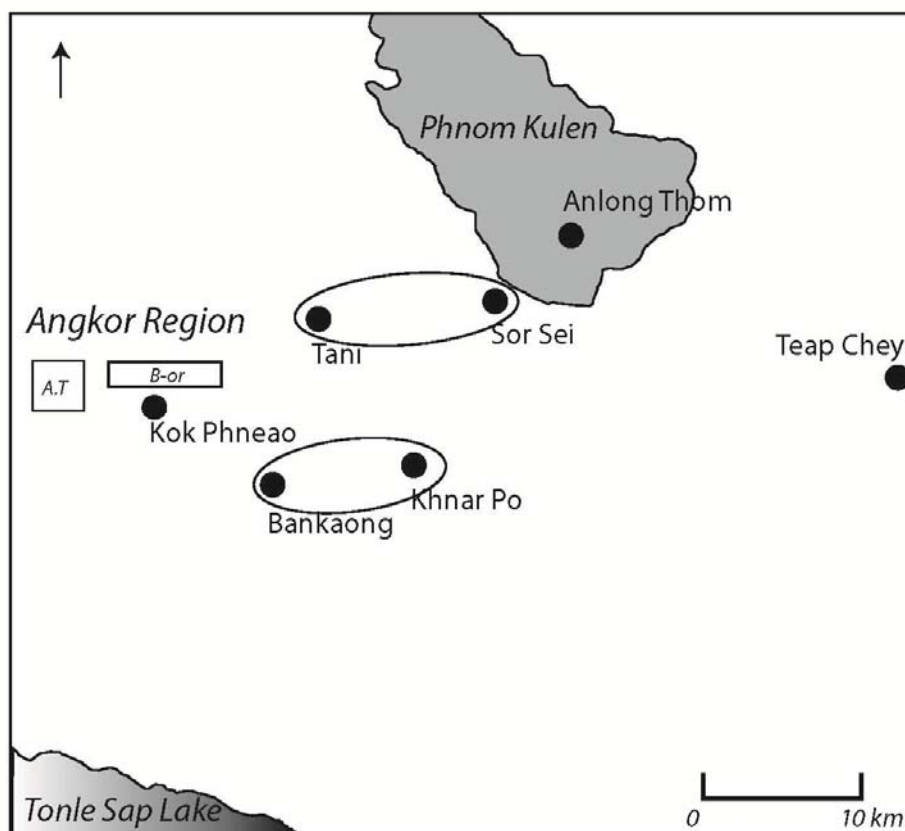


Figure 10: Relationship of unglazed stoneware and earthenware productions projected onto basemap.

Brown-glazed stoneware

Additionally to green-glazed ware, some brown-glazed stoneware was produced in Anlong Thom, mainly urns and lids, similar to those made in the green-glazed fabric (Desbat 2008, 2009, 2010, 2011, 2012). The brown-glazed stoneware shows chemical compositions different from the green-glazed stoneware and forms another, compositionally relatively homogeneous group (Fig.11, table 1).

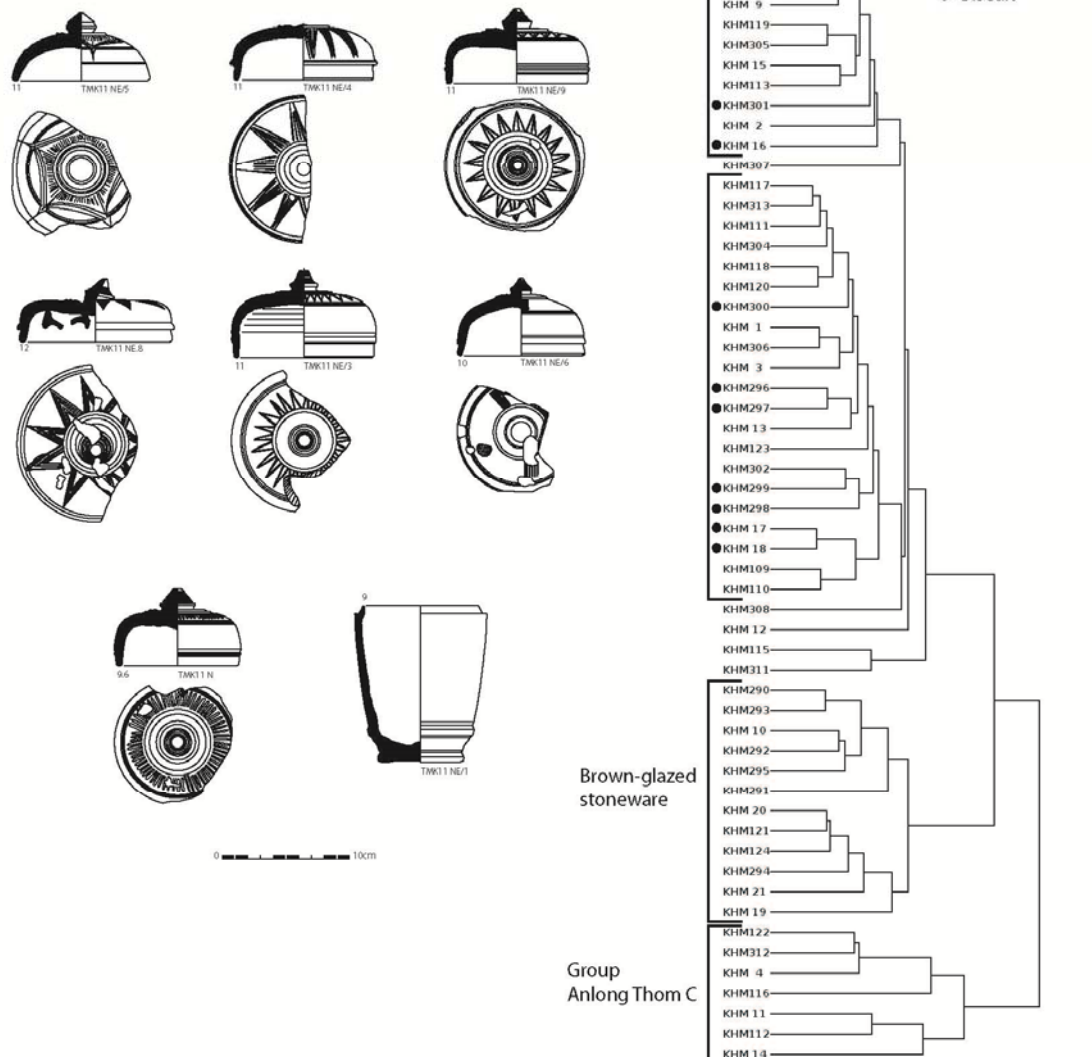


Figure 11: Some forms of brown-glazed stoneware from Anlong Thom and dendrogram of the cluster analysis of all analyzed glazed stoneware from Anlong Thom (calculated on 16 components).

Conclusions

The main goal, the definition of a solid reference database for stoneware produced in the Angkor region, has been realized, thus providing a useful tool for provenance determination of stoneware from consumer sites in future studies. Additionally, this study has addressed several issues. We suggest that the green-glazed stoneware from workshops in the Angkor region shows very similar compositions, and we can deduce the use of clay materials from the same geological origin. Two groups may be distinguished within this ware. Two groups were also distinguished for unglazed stoneware/earthenware, but the connections between workshops appear to be independent from the green-glazed groupings. Green-glazed and unglazed, as well as green-glazed and brown-glazed stoneware from the same workshop

show different compositions; for the reasons demonstrated, we deduce the use of a different raw material appropriate for each ware and not just different paste preparations. We also identified one homogeneous group for brown-glazed stoneware at Anlong Thom.

The possible reasons for the separation into two compositional groups are manifold, and include geological, geographical, or chronological variations, or a combination of these. We could evoke slight variations in the clay-beds at different locations; these may have been exploited by different workshops and/or at different periods. Perhaps the exploitation of resources was managed independent of the pottery workshops, and the separation reflects some kind of commercial or administrative connections. These possibilities are just some ideas of how these results concerning the workshops could be integrated into a larger concept of the Angkorian period. With these results, it is now possible to study the distribution of these angkorian stoneware productions on consumer sites.

Acknowledgements

The authors would like to thank the APSARA Authority who gave permission for us to analyse the samples, and special thanks to Chhay Rachna, who gave us the samples from the kiln site at Bankaong.

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Chemical compositions of unglazed stoneware and earthenware (values recalculated to 100 weight%). Listing in the order resulting from the cluster analysis (see dendrogram in Fig.8)

Analysis	Kiln sites	CaO	Fe ₂ O ₃	TiO ₂	K ₂ O	SiO ₂	Al ₂ O ₃	MgO	MnO	Na ₂ O	P ₂ O ₅	Zr	Sr	Rb	Zn	Cr	Ni	La	Ba	V	Ce	Y	Th	Pb	Cu
KHM383	Tani	0.40	6.73	0.831	2.46	69.21	18.40	1.46	0.0380	0.28	0.05	224	91	134	77	72	39	42	482	116	71	33	21	44	26
KHM377	Tani	0.44	6.56	0.853	2.36	69.87	17.94	1.47	0.0410	0.24	0.08	221	85	128	77	69	39	33	468	108	78	34	19	30	24
KHM211	Sor Sei	0.32	7.00	0.807	2.56	68.87	18.44	1.45	0.0628	0.28	0.06	234	76	145	191	67	41	43	467	115	74	32	23	35	32
KHM143	Sor Sei	0.48	6.62	0.814	2.89	67.74	19.15	1.72	0.0384	0.34	0.07	209	83	154	87	62	41	40	495	116	82	33	21	32	25
KHM 65	Tani	0.36	6.35	0.858	2.63	68.80	19.20	1.50	0.0841	n.d.	0.07	229	80	141	79	63	42	48	463	124	104	37	18	38	22
KHM203	Sor Sei	0.37	6.90	0.849	2.54	68.75	18.59	1.46	0.0508	0.28	0.06	225	74	142	84	59	42	47	428	112	95	34	22	24	29
KHM380	Tani	0.53	6.39	0.839	2.36	70.44	17.58	1.36	0.0714	0.23	0.05	229	70	127	75	67	47	57	538	107	97	36	19	42	26
KHM145	Sor Sei	0.28	5.48	0.813	2.53	71.15	17.62	1.52	0.0533	0.33	0.06	224	87	143	74	54	39	43	644	107	95	38	20	38	24
KHM146	Sor Sei	0.35	5.87	0.786	2.26	71.70	16.93	1.31	0.1447	0.42	0.07	233	74	126	75	56	42	44	523	103	103	37	17	45	30
KHM199	Sor Sei	0.30	6.20	0.815	2.36	71.10	17.29	1.39	0.1104	0.23	0.05	231	80	136	67	58	42	52	538	112	105	31	16	30	32
KHM 67	Tani	0.20	6.57	0.787	2.26	71.49	17.10	1.19	0.1708	n.d.	0.08	241	63	123	68	57	42	38	473	106	102	28	18	51	23
KHM207	Sor Sei	0.23	6.76	0.779	2.29	70.76	17.24	1.30	0.1364	0.31	0.05	237	68	134	65	59	42	45	446	109	89	30	14	31	26
KHM206	Sor Sei	0.32	7.15	0.796	2.51	68.99	18.21	1.41	0.1394	0.25	0.06	231	73	145	218	67	42	45	446	115	87	30	18	40	23
KHM209	Sor Sei	0.31	7.52	0.811	2.57	68.36	18.32	1.45	0.1674	0.27	0.06	228	75	144	74	65	42	41	538	125	107	32	21	55	28
KHM144	Sor Sei	0.44	6.99	0.798	2.81	67.35	19.14	1.73	0.1147	0.39	0.07	212	94	151	86	64	43	40	609	123	90	35	15	32	29
KHM200	Sor Sei	0.39	6.91	0.797	2.65	67.76	19.24	1.68	0.1234	0.24	0.05	221	81	145	87	66	56	45	457	111	104	29	19	35	26

n.d. = not determined

Chemical compositions of brown-glazed stoneware from Anlong Thom (values recalculated to 100 weight%).

Analysis	Kiln sites	CaO	Fe ₂ O ₃	TiO ₂	K ₂ O	SiO ₂	Al ₂ O ₃	MgO	MnO	Na ₂ O	P ₂ O ₅	Zr	Sr	Rb	Zn	Cr	Ni	La	Ba	V	Ce	Y	Th	Pb	Cu
KHM 10	Anlong T.	0.09	2.76	0.815	1.81	77.56	15.69	0.93	0.0572	0.05	0.09	328	41	100	66	54	44	62	332	71	97	44	16	146	20
KHM 20	Anlong T.	0.09	2.27	0.823	1.81	77.15	16.60	1.01	0.0170	n.d.	0.10	304	39	101	56	56	47	76	257	75	91	42	20	105	17
KHM 21	Anlong T.	0.13	2.45	0.851	1.66	77.55	16.08	1.06	0.0130	n.d.	0.08	337	39	93	75	57	40	41	250	82	80	55	17	150	14
KHM121	Anlong T.	0.09	2.45	0.818	2.14	77.18	15.96	1.05	0.0157	0.08	0.08	308	39	117	51	54	41	53	349	87	100	51	19	148	12
KHM124	Anlong T.	0.08	2.45	0.808	1.74	76.76	17.03	0.82	0.0205	n.d.	0.10	311	40	102	52	55	41	72	335	86	119	100	17	144	15
KHM290	Anlong T.	0.12	3.36	0.739	2.00	76.94	15.57	0.99	0.0621	n.d.	0.08	279	41	117	51	59	33	38	328	80	103	40	29	123	14
KHM291	Anlong T.	0.20	3.36	0.861	1.87	75.68	16.60	1.11	0.0675	n.d.	0.10	330	46	110	53	61	35	59	348	76	101	42	38	242	13
KHM292	Anlong T.	0.11	3.17	0.835	1.83	76.47	16.30	1.01	0.0532	n.d.	0.08	330	39	110	49	61	35	39	304	79	121	36	32	155	22
KHM293	Anlong T.	0.13	3.64	0.767	1.90	76.02	16.19	1.05	0.0658	n.d.	0.09	300	43	114	72	59	39	37	322	78	93	49	38	188	15
KHM295	Anlong T.	0.12	3.27	0.840	1.82	76.50	16.13	1.00	0.0835	n.d.	0.09	331	44	107	65	63	40	52	340	73	107	41	31	121	14

n.d. = not determined