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Geochemical analyses result of prehistoric pottery from the site of Tol-e Kamin (Fars, Iran) by pXRF

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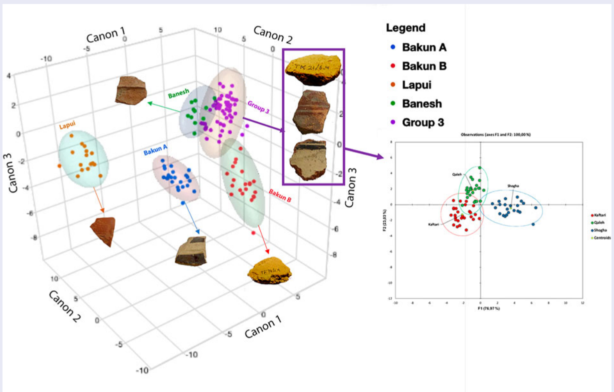
ABSTRACT
A series of pottery samples from the Iranian site Tol-e Kamin, ranging from pre-historical period to the New Elamite, were analyzed in order to study the geochemical variability of the pottery assemblage. A total amount of 168 measurements were obtained using a portable XRF device and were statistically handled. The results could successfully distinguish the geochemical composition of potteries from the chalcolithic to the New Elamite periods in the Kur River Basin. A major shift in the use of different clay sources could be detected since the Proto Elamite period and afterward, in which the carbonated and marl content clays represented by Ca, Ba and Sr shifted to clay sources with a tendency to non-carbonate silty clay Al, Ti, and Fe from a different geological background. The results stress the importance of further provenance studies to address issues of trade and exchange possibilities in southwestern Iran.



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Portable XRF; Kur River Basin; archaeology of Fars; pottery analysis; DA; PCA

Introduction

Recent studies have reasonably discussed the potential of chemical analysis of archaeological pottery as an important material culture in deriving of socio-cultural information of ancient societies (Arnold 1999; Hughes 1981; Noll and Heimann 2016; Rice 2015; Shepard 1957). In an archaeological site with intensive settlement phases like Tol-e Kamin, a chemical analysis of the pottery assemblage can deliver insights toward preference, choice and change of composition through time. During an exploratory survey conducted by Goethe-University and Iranian Center of Archaeological Research in summer 2017, around 1100 sherd were collected associated with the known archaeological periods (Rajabi and Wicke 2017). Among them, a series of representative prehistoric sample was selected from the Chalcolithic up to New Elamite periods for chemical analysis. In a first step, it was intended to conduct a pilot study in order to evaluate if the variability between different temporal clusters is significant enough that could be detected by portable XRF. As a second step, it was intended to define to what extent archaeological periods overlapped with geochemical clusters for further classification studies. Few previous archaeometrical studies on ceramic in the region were carried out either on archaeological periods across different sites (Blackman 1988; Pincé et al. 2016, 2018, 2019) or on individual periods at one or more sites (Alden and Minc 2016; Blackman 1981). In this study, pottery samples from several archaeological periods collected at the site of Tol-e Kamin are studied, in order to provide a better understanding of the diachronic dynamics of geochemical change in ceramics from the Kur River Basin (hereafter KRB). The results are critical for a further classification of the regional pottery and provenance studies in the future.

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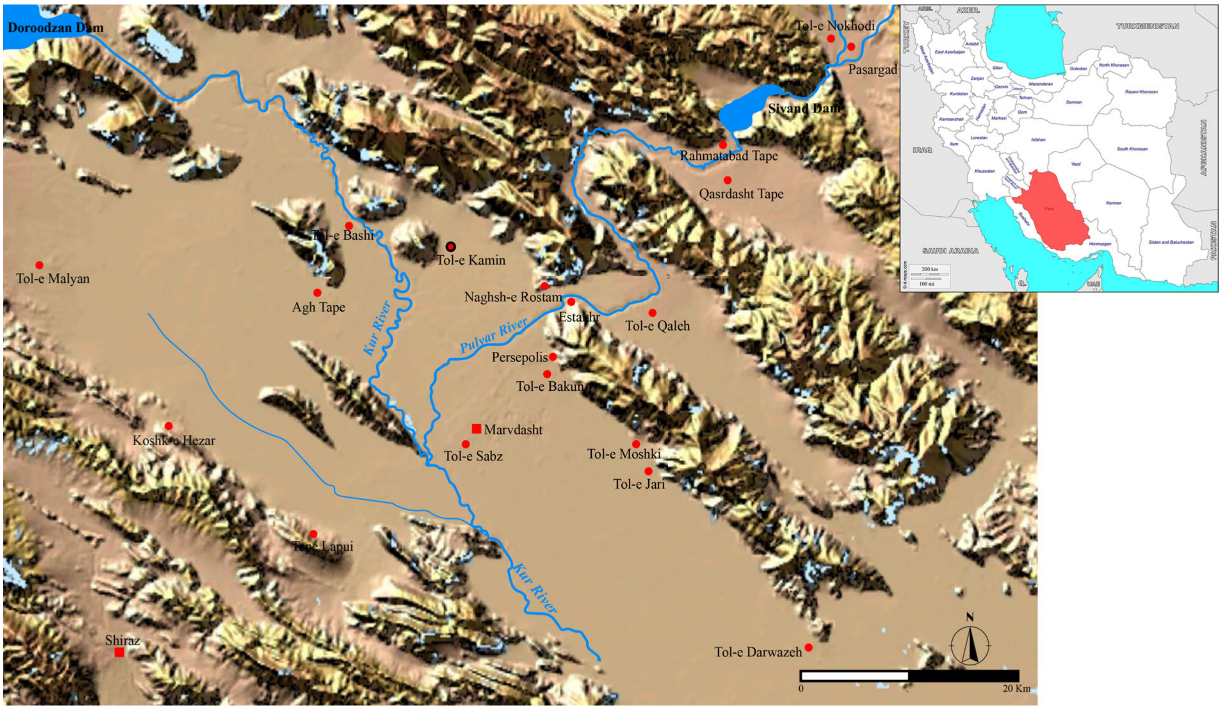


Figure 1. The geographical location of Fars Province in Iran (top right corner) and Tol-e Kamin in relation to other important archaeological sites are illustrated in this topographic map. Source: German Aerospace Center (DLR).

Geography and archaeological location of Tol-e Kamin

Tol-e Kamin is located to the northwest of the famous site of Persepolis in southwestern Iran in the province of Fars. The highest point is located at 52°48′57″ (East), 30°1′00″ (North) and approximately 1620 m above sea level. It is a flat but wide settlement mound in the plain of Marvdasht inside the KRB. Despite its rather flat appearance with a height of only about 9 m, it is one of the more prominent sites in the area, covering about 260 × 330 m (Figure 1). In 1952 and 1955, the Belgian archaeologist Louis Vanden Berghe undertook small soundings on the mound and discovered three tombs, dated to the New Elamite Shogha/Taimuran phase, that is the second half of the second millennium BC (Overlaet 2007). William Sumner and Linda Jacobs later surveyed the site and noted in particular a strong prehistoric occupation, although the surface pottery includes Islamic pottery as well (Sumner 1989a, 146). Our recent survey discovered the main concentration of pottery at the center of the mound and along the fringes; a further dense scatter of Sasanian style pottery was visible at its SW-corner. Bronze Age pottery, that is Banesh and Kaftari material, as well as pottery dating to the Qaleh period dominates the main mound, Related sherds were discovered almost all over the mound, which argues for a broad occupation of the site from the fourth millennium onwards. Middle to New Elamite material was more widely scattered with a little predominant occurrence along the western part.

Materials and methods

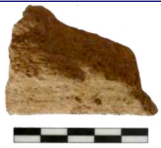
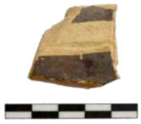
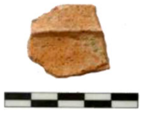
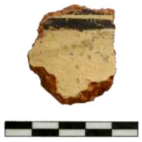
Ceramic samples

During the above-mentioned fieldwork, a representative on-site survey was conducted that yielded 1140 single sherds, which were recorded and macroscopically determined according to styles and wares. A series of 64 sherds, representative of the various periods, were exported and analyzed in the Frankfurt laboratory for Ceramic Studies. The pottery sherds are ranging from coarse, handmade chaff tempered to very finely sand tempered wares. The typical macroscopic and archaeological characteristics of the analyzed samples are summarized in (Table 1).

Portable X-ray florescence

Among the various analytical methods, handheld portable XRF has gained increasing attention and has shown different aspects in the archaeological studies (Shackley 2014). Especially regarding to a remarkable development in Silicon Drift Detectors (SDD), many archaeological studies could successfully help to define issues of composition or answer questions on the provenance of pottery (Ceccarelli et al. 2016; Goren, Mommsen, and Klinger 2011; Helfert 2010; Rahimi Sorkhani and Eslami 2018). Being non-destructive, time and cost-effective, and achieving relatively good results makes pXRF analyses an easy to use technique for fieldwork in archaeology (Liritzis and Zacharias 2011, 112). Essential factors such as calibration, sampling strategy as well as nature the of samples have to be respected, of course, in

Table 1. Macroscopic description of representative samples analyzed in this study. Number of analysis presents counts of analyzed batches. Each batch has been repeated for three times in order to reduce bias error as much as possible.

Period	Macroscopic description	No. of analysis	Appearance	Relative dating (BC)
Bakun B	Medium to coarse grain, handmade with short chaff tempers, partially grit, light to dark brown body	$n = 20$		5200–4800
Bakun A	Pale cream body with buff slip, almost no temper to fine sand grain, black decoration on the surface	$n = 24$		4500–4100
Lapui	Fine wheel made, dark brown to sharp red body, burnished slip without decoration, almost no temper, few fine lime grains	$n = 28$		4000–3500
Banesh	Medium to fine grain, pale red to orange body, tempered with mud rock and girt particles	$n = 20$		3500–2800
Kaftari	Medium to coarse grain, tempered with few grit and sand inclusion, pale red body with grayish cream slip, black decoration on the surface	$n = 24$		2200–1600
Qaleh	Medium to coarse grain, orange body with buff slip, black decoration, fine grit and sand grain inclusions	$n = 24$		1600–1300
Shogha	Fine to medium grain, dark brown to grey body, red decoration, mixture of chaff and sandstone tempters	$n = 28$		1300–900

order to obtain meaningful results (Forster et al. 2011; Tykot 2016).

Pottery sherds in this study were analyzed with a Portable Energy Dispersive X-Ray Florescence (ED-XRF), from Niton XL3t 900S; GOLDD+ (Geologically Optimised Large Area Drift Detector) technology model. X-ray was generated by a 50 kV and 200 mA tube, and the secondary radiation is detected by a larger silicon drift detector (SDD). The elements are detected by TestAll Geo Mode in order to cover the essential elements of interest in ceramics. This Mode uses both the Compton Normalization and the Fundamental Parameters calibration in order to minimize bias error effect of the results. Three measurements have been carried out on each freshly broken pottery sherd with the most effort to have the analyzing surface as flat as possible. This procedure is recommended to reduce possible errors of heterogeneity in measurements and consider as much inclusion as possible (Forster et al. 2011; Poupeau et al. 2010). Each point was radiated for 300 s with different intensities according to the filters used for low, medium and heavy elements. In this case, three 60 s irradiation time set for the standard, high and low z elements respectively. An extra 120 s also was performed for light elements.

Before and after each batch of measurements, a standard sample DIN-NCS DC 87102 for silicate soils were measured to control the accuracy of the setting. The values in bracket are presenting relative standard deviation RSD% in percent for 30 measurements. Elements with a concentration higher than LOD (three times of standard deviation) and less than 10% RSD have been subjected for statistical tests. Si (2.1), Ti (1.2), Al (3.9), Fe (1.9), Mn (3.9), Mg (22.1), Ca (7.0), K (2.6), P (21.88), V (8.7), Cu (5.8), Zn (6.2), Rb (2.2), Sr (2.01), Y (2.06), Zr (2.07), Nb (4.27), Ba (2.8), Pb (5.7) and Th (7.9).

We omitted the Si from the data set, in order to eliminate the impact of large sand inclusions for some elements. Because Si has a dominant statistical

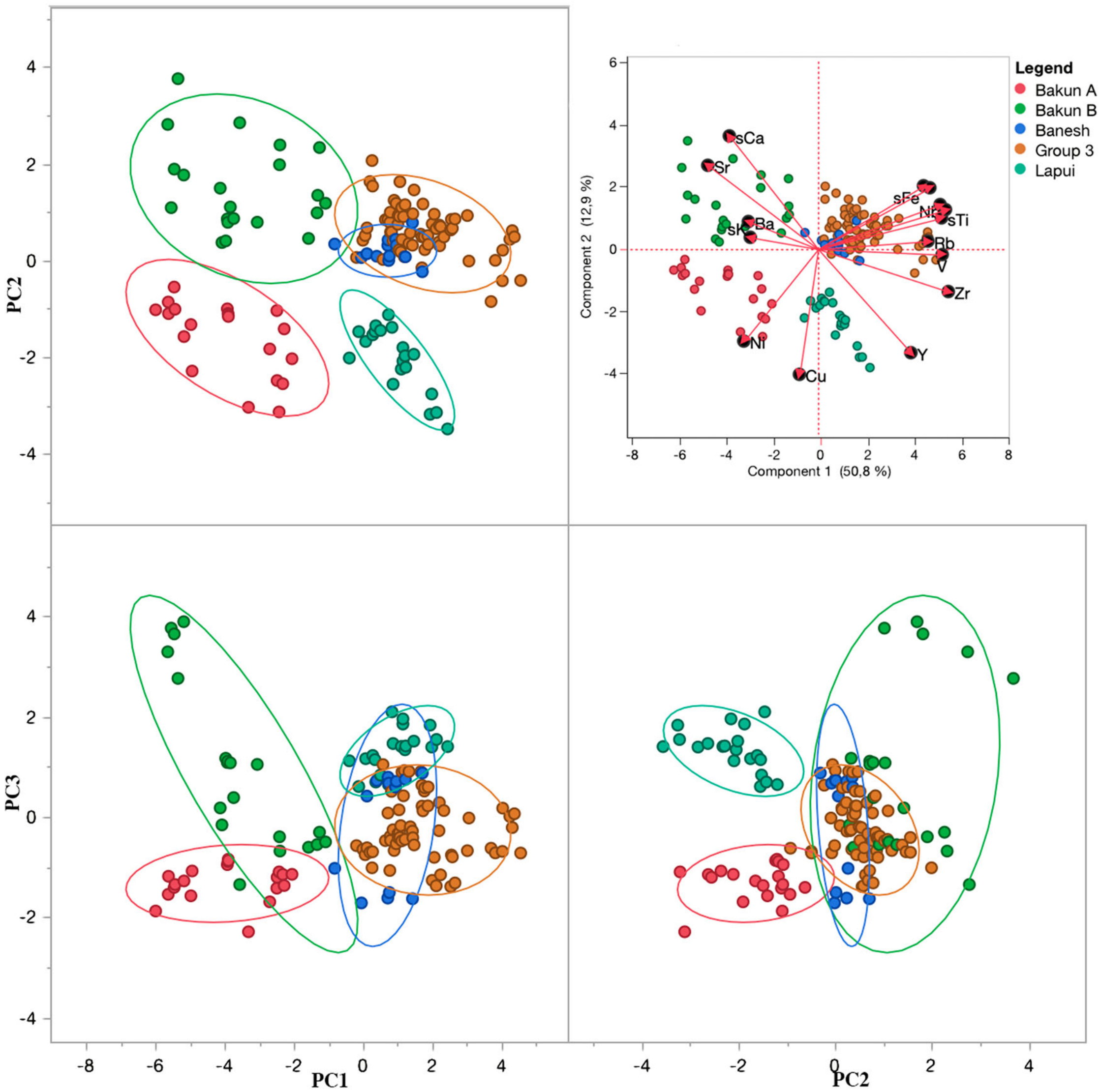


Figure 2. PCA scatterplot for first three main components. A series of main (Ca, Fe, Ti and K) and trace elements (Sr, Ni, Cu, Y, Zr, V, Rb and Nb) represent a clear separation of samples from different archaeological periods. Confident ellipse has been calculated with 90%. Bakun A, B and Lapui are separated primarily by first and second components, while the Banesh and Group 3 (Kaftari, Qaleh and Shogha) correlate with third components.

weight, it could negatively affect the statistical tests (Emmitt et al. 2018). On the other side, Mg, P and S were also omitted due to their high deviation. Especially P and S can be easily affected by a post-depositional process during and after burial and due to their high concentration will consequently affect the trace element content (Freestone 2001; Pillay et al. 2000). The obtained values from the device were calibrated through the coefficient factor of each element. These factors have been obtained through the internal calibration process in Ceramic Research center Frankfurt University (Helfert et al. 2011; Helfert 2013). total of 168 measurements in combination of major, minor and trace elements including Ti, Al, Fe, Mn, Ca, K, V, Cu, Zn, Rb, Sr, Y, Zr, Nb, Ba and Pb have been subjected to statistical tests. Statistical calculations have been performed by Statistical Discovery JMP 14.0.0.

Results

The Principal Component Analysis (PCA) were able to explain 50.8%, 12.9%, and 9.5% of all variables on first, second, and third significant components, respectively. The Kaiser–Meyer–Olkin measure verified the sampling adequacy for the analyze, KMO = 0-77 indicating Principal component test is suitable for this dataset. Bartlett's Test of Sphericity (approx. Chi-square = 2924.94, df = 136, $p < 0.005$) indicated that correlations between items were sufficiently large for PCA. Three components were extracted with eigenvalues 7.6264, 1.9285 and 1.4374, respectively represent 73.28% of all variance.

The first three main components are illustrated on a Scatter matrix plot in order to reveal the correlation pattern of each pottery group (Figure 2). At the first look, on the first component PC1, Bakun A and B assemblages share similar composition and are

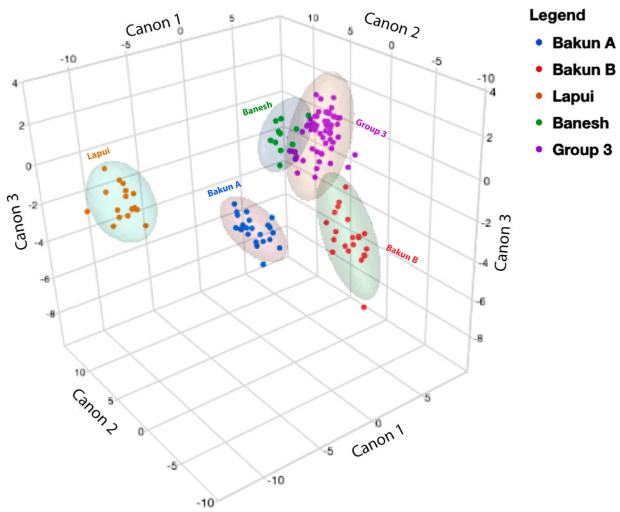


Figure 3. DA analysis for the same elements in order to estimate the significance of clustering. The results are illustrated in a 3D diagram including three canonical factors. Bakun A, Bakun B and Lapui have been significantly separated based on three factors.

distinguished from the others due to the higher calcareous components. This result is also followed by a higher amount of Sr and Ba for both groups of assemblage. because of coarse texture of Bakun B with inhomogeneous chaff-tempered fabric the composition of samples varies even within the group. On the other side, pottery from Group 3 (Kaftari, Qaleh, Shogha ware), Banesh, and Lapui periods were overlapping on the PC1 and positively correlated with Fe, Al, Ti, Rb, V, Zr and Nb. This group of pottery is distinguished from each other primarily in terms of PC2 and PC3. The clay in Lapui group, for instance, contained a lower concentration of Cu, Y and Ni, whereas the clay in group 3 is more abundant in K, Rb elements.

Discriminant Analysis (DA) based on standard linear covariance for the same element assays like PCA have been performed in order to examine the significance of dissimilarities between determined assemblages. Three canonical discrimination factors with eigenvalues 36.24, 14.12 and 2.69 calculated from the overall pooled within-groups covariance matrix, which significantly discriminants 67.36%, 26.25% and 5.006% of all covariance samples (df = 472.05 < 0.001) respectively. The result of the DA test is illustrated on 3D scatter plot with 90% confidence ellipse for each assemblage (Figure 3). In the first canonical factor elements of K, Ca, Fe, Al, Rb and Sr significantly variates whereas on the second factor primarily trace elements namely Zn, Zr, Nb, Pb and V differentiated. Calculated Canonical factors in Centroids for this assay of elements demonstrated that Bakun wares and Lapui are significantly different from Banesh, and Group 3 according to all three canonical factors.

Group 3 is composed of three types of assemblages belong to the middle to the New Elamite. These are Kaftari, Qaleh and Shogha wares. Confusion matrix for the cross-validation values indicated close similarity for these group of pottery around more than 50% in composition (Table 2). However, dissimilarity for such variables can be underestimated under the statistical weight of other groups with large eigenvalues. In order to study these groups in more details, distinguished groups in the previous test have been omitted, and the only assemblage from Kaftari, Qaleh and Shogha are subjected to DA (Figure 4). Two discriminant factors explain 76.97% and 23.03% of all variance. However, only the first factor (df = 118 p < 000.1) is significantly discriminant in the observation. i.e. in this case, Shogha wares differentiated from the other groups, as it contains a higher amount of Zr, Ba and Fe, and less Nb, Rb and Ni.

Discussion

In the research reported here, we investigated how variable are the pottery assemblages from different archaeological periods at Tol-e Kamin in the KRB. This preliminary result aims to evaluate the pottery composition change within a single site diachronically. The analysis detects the calcareous composition of Bakun A and Bakun B wares. Bakun B (5400–4800 BC) with coarse painted handmade pottery has been characterized as transitional between Neolithic Jari and late chalcolithic Bakun A (Sumner 1977, 300). Bakun A (4500–4100) with distinctive fine black decoration on buff has been considered as the beginning of more complexity in society and pottery production organization in KRB (Alizadeh 2003, 2006; Sumner 1977). Despite a visible difference of Bakun B and A, chemical results showed that both wares are correlating with Ca, Sr and Ba, which is representative for a calcium-rich clay sediment. Tiny lime pebbles can also be visually observed. Among them, strontium is a geochemically stable element and tends to remain in sediments even during transportation and sedimentation. It works as

Table 2. Above: The table show statistical distances for canonical factors calculated by statistical DA test (only distance of the three first factors are significant). Bottom: Confusion matrix measurements present the percentage of correctness for classified groups.

Canonical factors in centroids						
	F1	F2	F3	F4	F5	F6
Bakun A	−6.26	−4.21	1.24	0.18	0.09	−0.01
Bakun B	−1.57	0.29	−2.21	1.51	0.47	−0.06
Lapui	−2.82	5.00	1.06	0.12	0.02	0.25
Banesh	2.02	−1.34	−0.66	0.08	−1.15	0.77
Group 3	4.27	−0.48	0.56	0.03	0.20	−0.18

Confusion matrix for the cross-validation results									
from \ to	Bakun A	Bakun B	Banesh	Kaftari	Lapui	Qaleh	Shogha	Total	% correct
Bakun A	24	0	0	0	0	0	0	24	100.00%
Bakun B	0	18	1	1	0	0	0	21	85.71%
Lapui	0	0	0	0	28	0	0	28	100.00%
Banesh	0	1	16	1	0	2	0	20	80.00%
Kaftari	0	2	0	15	2	4	4	27	55.56%
Qaleh	0	0	1	8	0	11	4	24	45.83%
Shogha	0	0	4	1	0	5	8	18	44.44%

a proxy of calcium and has been successfully used as an indication of detrital limestone/packstone, reef limestone, and micritic clay deposits such as marls (Morgenstein and Redmount 2005, 1621). It might be an insight into the application of alluvial clay in the manufacture of pottery. Preliminary petrographic study on Bakun ware from mamasani district in Fars also confirmed calcareous containing small grains of calcite and quartz in the manufacturing of Bakun ware (Petrie et al. as cited by Weeks, Petrie, and Potts 2010, 254). Considering technical advancement and complexity in decoration and production of Bakun ware A, Sumner has suggested that likely specialized potter was responsible for the production of Bakun ware (Sumner

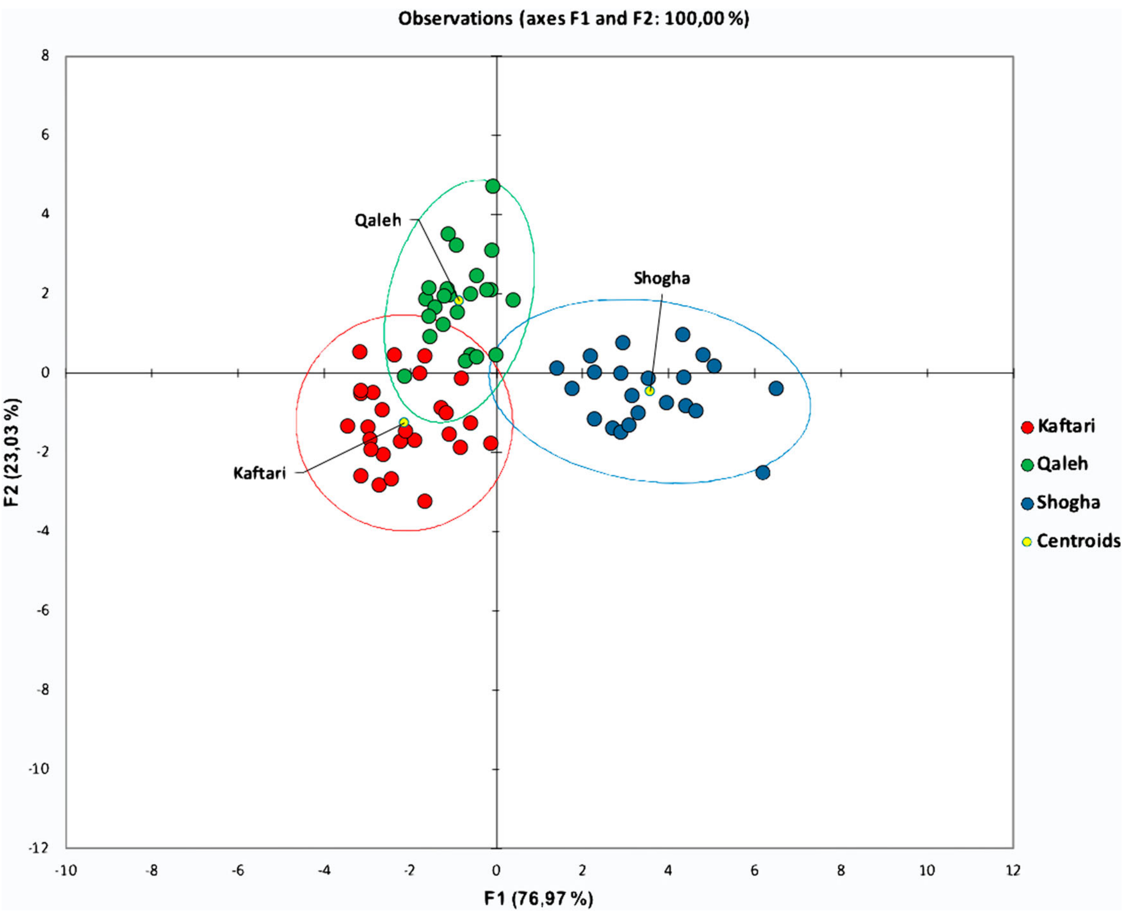


Figure 4. DA analysis for pottery in Group 3. Shogha sherds are separated based on the first main component presenting different clay source composition, while the other two periods are distinguished by second main components. Confident ellipse was calculated with 90%.

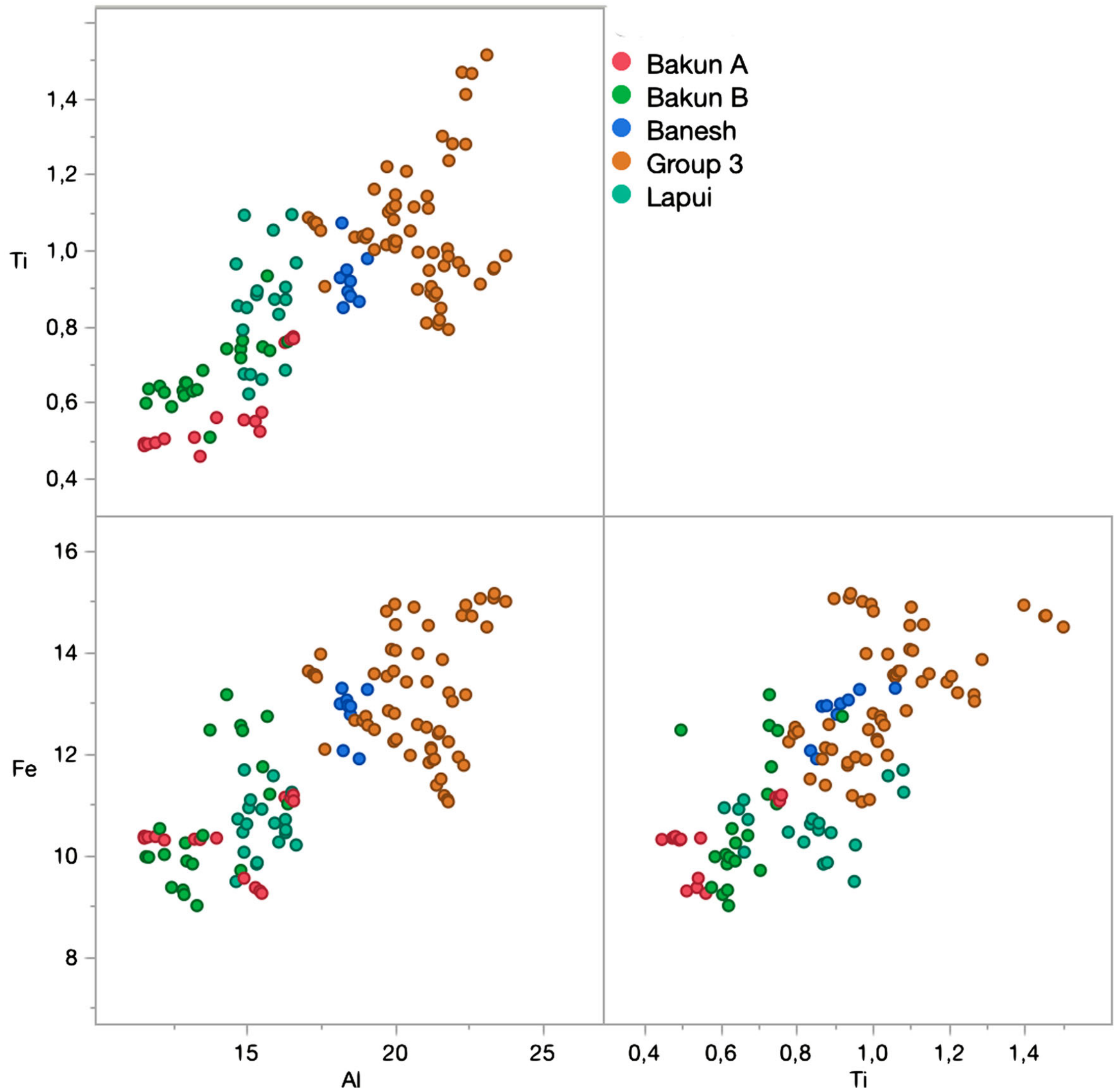


Figure 5. Scatter matrix plot for three main elements Ti, Fe and Al. Bivariate graphs show two main compositional groups associating with three elements. Confidence ellipse was calculated with 90%.

1994, 59). Given skilled craftsmanship and absent of pottery production evidence in the majority of Bakun sites, raised the possibility that specialized Bakun-period ceramic production directed toward local exchanges of pottery taking place within parts of the KRB and other relatively small and self-contained regions such as the mamasani district (Weeks, Petrie, and Potts 2010, 265). The distinction from Bakun A wares has also been shown recently in the chemical composition of this ware from Tol-e Gap (Pincé et al. 2016). Pincé detected more intensity in Cr peaks, and our result revealed more concentration in Ni. However, both elements are low to mid Z elements and can be found in correlation to each other semi-quantitatively (Hunt and Speakman 2015, 630). Given the small quantity of recovered Bakun ware sherds (5%) found in Tol-e Kamin and a relatively short distance to Tol-e Bakun Site (9 km), it could be likely that Bakun ware in Tol-e Kamin was an exchange ware rather than local production. However, a detailed technical study on Bakun Ware, in general, is necessary to confirm this statement. Based on the same elemental correlation for Bakun A and Bakun B wares it can be suggested that likely similar sedimentary depositions have been used for production process, However, the preparation process for both wares must have been different, which indicates other composition preferences in the preparation and processing of clay (Tite 2008).

At the beginning of the fourth millennium BC, a characteristic burnished red ware labeled as Lapui ware appears in several sites of the KRB. The geochemical composition of Lapui sherds from Tol-e Kamin demonstrate a significant difference comparison to the older Bakun wares. Previous technical study on ceramic material from the KRB detected a shift in usage of low Ca content clay in Lapui period (Blackman 1988, 106). Our analysis, in addition to that, demonstrates a positive correlation of K, Rb representative of feldspars and mica group clays. This result shows a tendency toward employing different clay sources in the production process of Lapui pottery, which is an

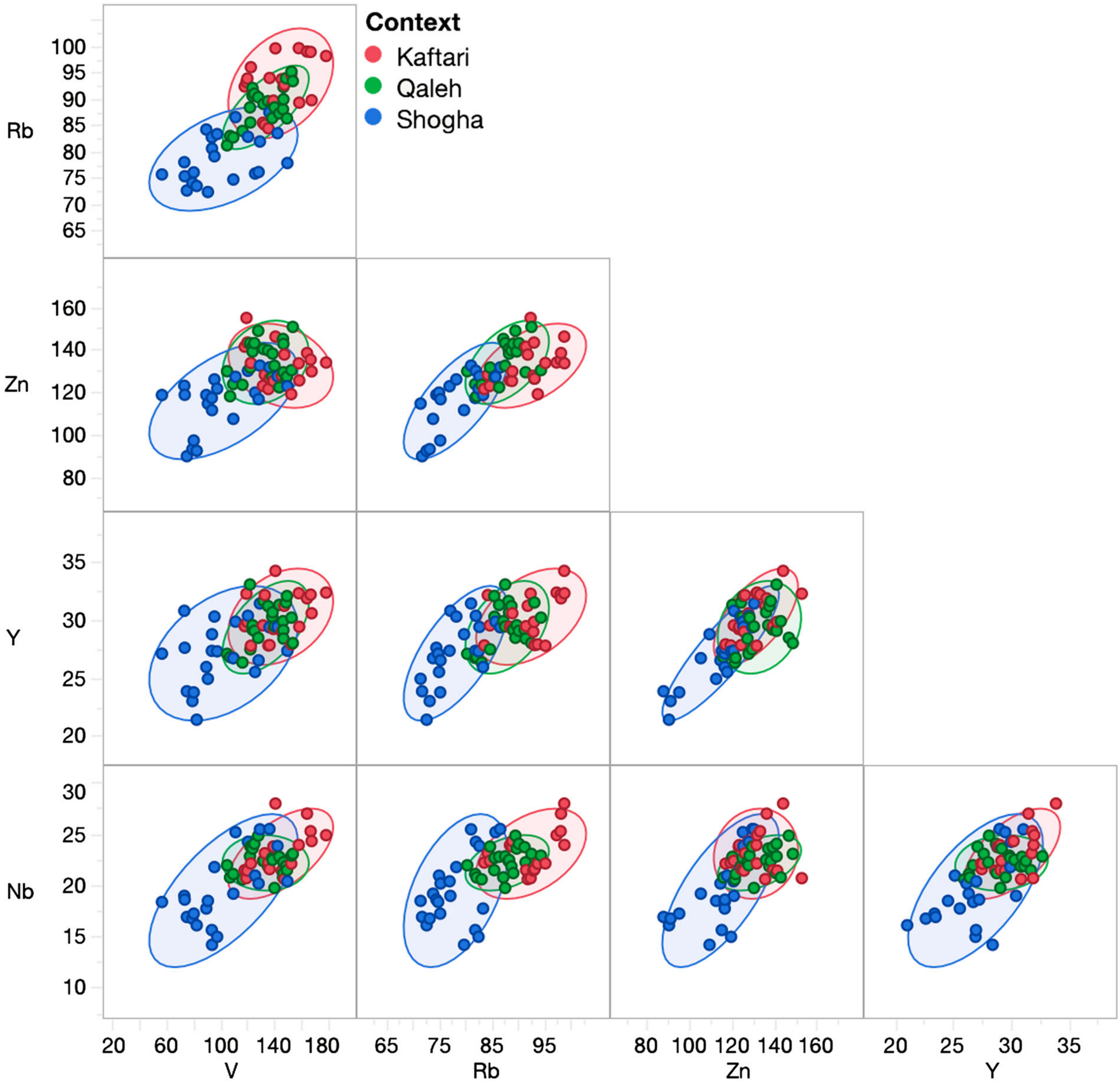


Figure 6. Bivariate plots for significant trace elements representing different concentrations for Shogha pottery in comparison to the Qaleh and Kaftari periods. On the other side, the earlier periods are overlapping regarding to several element. Confident ellipse was calculated with 90%.

insight to the usage of initial Mudstone clay pallet. Sumner suggested a shift in the spatial pattern of settlement and proposed a change of lifestyle towards pastoral mobility in the Lapui period (Sumner 1988, 1989b). More contact with other cultures, especially in southwest Iran, was proposed as a reason for this stylistic change rather repopulation in the region (Blackman 1988, 104). Still, the archaeological evidence reports a co-occurrence of both black on buff (Bakun A) and fine plain red ware (Lapui) in several parts of the KRB (Alizadeh 2006; Potts et al. 2009). The recent archaeological survey and excavation in Tape Maher Ali, e.g. revealed no sharp break between the pottery production between Lapui and Bakun A, but archaeological strata containing both wares together (Sardari 2013, 202). The co-occurrence of both types of Bakun A and Lapui wares in the same archaeological context proposes exciting insight into the bilateral origin of clay sources. the potters have applied different raw materials, which might indicate different technological or ideological preferences regarding pottery function and use.

A particular pattern could be detected in our analysis, distinguishing higher content of Fe, Ti followed with Al for Banesh and "Group 3" samples, in contrast to earlier periods (Figure 5). The Banesh pottery analyzed in this study is the characteristic Banesh grit-tempered ware, which can be slipped and decorated with black, white, and red paint (Abdi 2004, 259). Technological change from fine-red Lapui pottery to coarse grit-tempered pottery was interpreted as a local response to the new socio-economic need of large storing jars in the Banesh Period (Blackman 1988). specialized production factors have been identified for this kind of pottery in the KRB in compare to chaff-tempered pottery (Alden 2003). In this regard, Alden has identified similar clay sources with high Al content for the majority of Banesh period sites in KRB (Alden and Minc 2016, 868). Another chemical and mineralogical study on grit-tempered pottery from the Banesh period has revealed that Possible sources for the clays used to produce grit-tempered Banesh pottery could be shale beds in the folded mountains above the talus slopes rather than alluvial sediments (Blackman 1981). Recent chemical and mineralogical study on KRB pottery of middle to the late second millennium BC showed Fe, followed by K and Ti, appeared to be the most prominent elements (Pincé et al. 2019). In the absence of petrography, it is not possible to confirm specific origins of clay in Tol-e Kamin, but the our results show a remarkable shift in clay composition since Banesh (Proto Elamite) period.

DA statistical test only on "Group 3" samples revealed more clusters for three type of wares (Figure 4). Whereas Qaleh and Kaftari samples presented more overlapping regarding similar elemental composition, Shogha samples are separated from the rest. A series of bivariate associated with main discriminant elements Zn, Rb, Nb and Y clearly showed this similarity (Figure 6). This graph demonstrates a likely similar clay source for Kaftari/Qaleh in comparison to Shogha wares. The Kaftari period begins around 2200 BC with an increase in the number of settlements (Sumner 1989a). Pottery in the Kaftari period is coarse and handmade, which is also visually similar to Qaleh ware; however, the decoration on the surface is slightly different. Qaleh ware developed from kaftari and they were partially contemporary until 1600 BC but the transition between both phases is not clear yet, and more fieldwork is needed to clarify the matter (Haerinck and Overlaet 2003, 194). Qaleh ware also overlaps with ceramics that define the KRB Shogha/Taimuran phase, starting sometime between 1600 and 1300 and lasting until c. 900 BC or possibly even later (Overlaet and Pincé 2018, 5).

Recent mineralogical analysis on pottery of these two periods from different sites confirmed that Qaleh and Shogha wares were produced from significantly different raw materials. Pincé analyses Qale Ware, fine middle Elamite, and clay sources from the different spots of river stream and Zagros foothills and suggested that local, alluvial clays were in general systematically used for the production of these wares. On the other side, her result also showed that Shogha ware from different sites, including Tol-e Kamin, have similar composition more affiliated with mountain foothills (Pincé et al. 2019, 563). Our analysis of pottery from Tol-e Kamin also confirmed that a separation of Qaleh and Shogha Ware, based on a different raw material, could not only be observed between sites (as Pincé showed) but also within a single Site. Given the archaeological evidence from Tol-e Kamin and the number of recovered sherds from kaftari and Qale ware (30%), probably these types of pottery have been also produced locally in Tol-e Kamin. However, a detailed mineralogical analysis should be carried on exclusively on pottery from Tol-e Kamin to be able to confirm this statement. In the scarcity of technical studies on pottery production in Fars province, this study is a begin for more systematic chemical and mineralogical investigations on pottery of Tol-e Kamin to fill the gap of various pottery ware production.

Conclusion

Our main objective in this study was to identify the extent of geochemical compositional variability for pottery samples obtained from different archaeological periods in Tol-e Kamin. In this case, the calibrated portable XRF devise with the aim of statistical test of PCA and DA was able to detect several geochemical clusters. The results confirm that the samples are not merely distinguished macroscopically, but also geochemically followed various criteria for the selection and application of resources. Although Bakun A and B ware show more correlation with Ca, Ba and Sr, representing for detrital limestone, and micritic clay deposits such as marl, Lapui is distinguished with higher alkali metal elements including K and Rb. Given archaeological evidence and the number of recovered sherds, a non-local origin for pottery of Bakun and Lapui is likely. On the other side, in some cases, change in composition of clay seems to coincide with socio-economic changes like in the transition from Lapui to Banesh or from Kaftari/Qaleh to Shogha/Taimuran wares. A considerable change in the composition of clay could be detected since the Banesh period in comparison to earlier periods. The grit-tempered Banesh pottery and proceeding periods of Kaftari, Qaleh, and Shogha contains a significantly higher amount of Al correlating with Fe and Ti. The change in the composition of Banesh pottery has been related before to the need of production saving jars. Our analysis also showed a significant difference among Kaftari /Qale and Shogha ware, including different trace element concentrations, namely Rb, Nb, Zn, Y. According to results, Kaftari and Qaleh ware in Tol-e Kamin have shared similar local source clay since the majority of pre-Islamic sherds recovered in Tol-e Kamin belongs to these periods. From the current point of view, the establishment of a geochemical reference database for clay and ceramics from the KRB is necessary in order to be able to address issues of the provenance of pottery particularly in the cases of chronologically neighboring periods. Despite of vast diversity of pottery manufacture tradition in Fars Province, still technical studies on pottery stand at the beginning. in the next stape, chemical and mineralogical analysis of characteristic periods in KRB should be performed on order to fill the gap of data on technical issue.

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No potential conflict of interest was reported by the author (s).

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