

The Case of the Mycenaean Site of 'Kastrouli' Near Delphi; Characterization of Pottery and Clay Material: A First Assessment of the Results through XRF and XRD Analyses

Tonia Tsourouni*

Archaeologist MA, University of Athens, Greece

*Corresponding Author: Tonia Tsourouni, Archaeologist MA, University of Athens, Greece

ABSTRACT

The theme of this prototype research concerns the analysis of selected pottery fragments (K1-K81) derived from the first excavation period of the prehistoric site "Kastrouli" in the neighboring area of Delphi. Analyses were performed on the samples by the methods XRF and XRD (along with Munsell color system) in order to identify the chemical and mineralogical elements that lead to the characterization of the clay used for the manufacture of ceramics. Moreover, analyzes were performed in clay soil samples (DS1-2-3-4), collected from surface survey in the neighboring areas of Agia Irini, Limnos and Meteles in order the local raw material to be examined. In this paper, are presented the results obtained from the analysis that focus on the chemical composition of both the clay and the raw materials. The results concern the characterization and the provenance of the pottery under study, along with the raw materials manufactured. The contribution of this study leads to the identification of the pottery and the long-term interpretation of the technology, applied by the potters at this Mycenaean site, concerning the technical level and the origin of the clay.

Keywords: Mycenaean pottery, clays, XRF/XRD methods, Prehistoric site, cluster analysis

INTRODUCTION

The subject of this paper, as well as all the recordings and analyzes for this purpose, were carried out in the frame of the author's dissertation on the acquisition of her master's degree. The selected pottery that was under study comes from the excavation research of the prehistoric site "Kastrouli". This Mycenaean site (~ 1,300 BC) is located near the village of Desfina, just 10 km away from Delphi, in the wider area of the Prefecture of Fokidos (Fig. 1).



Figure1. The location of the site on the geomorphological map and its proximity to other important positions (via Google earth)

It is notable that it is close to Antikyra (5km to SE) which seems to be directly related to its port in the prefecture of Fokida in antiquity (Sideris 2014, 24-26, 29-31), as well as to Itea. It is a Mycenaean site located on a hill (tuba), at an altitude of about 700m. (Fig. 2).

The hill has a predominantly limestone substrate and it is noted that limestone remains can be seen throughout the hill area. This strategic position refers to the corresponding case of Mycenae in the Peloponnese, during the same period.



Figure2. The Mycenaean site "Kastrouli" by aerial photography via Google earth

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It is noteworthy that the fortification of the Cyclopean system in which at least two construction phases are observed is well preserved (Sideris et al, 2017, 282). The wall of the place has a maximum diameter of 160m. on the B-N axis, while at the same time indicates the importance of the position (Fig.3).



Figure3. NW view of the fortification with the two construction phases obvious

The samples that were analyzed are included in the findings of the first excavation period that took place during the summer period from July 20 to August 3, 2016. Specifically, an archeological-archaeometric survey was carried out at "Kastrouli" as at least three Mycenaean tombs have been identified within the wall and there are indications of building remains.

The archeological excavation was led by Dr. Athanasios Sideris in collaboration with Prof. Thomas Levy of the University of California San Diego with the participation of students. The archeological research was conducted by Prof. Ioannis Lirintzis of the University of the Aegean. Prof. G. Tsokas (AUTH) also participated in the geophysical survey and Prof. A. Georgopoulos (NTUA) in the geospatial system. On behalf of the Ephorate of Antiquities of Phocis, the archaeologist Mr. Anthoula Tsaroucha was responsible.

Systematic research had focused on the looted - in the past - Tomb A, which is formed by large stone slabs on both sides while the road ends in a carved lateral part of a stone substratum that underlines a type of mixed chamber and stone tomb (Sideris et al, 2017, 280). At the same time, excavations were carried out in two sections that were opened for research, in the wider area around the Tomb.

Findings in general include Φ and Ψ type figurines, pieces of gold jewelry, animal bones and several human bones that form skeletons (side burials). In terms of micro-craftsmanship, mainly bone beads and various metal finds were collected.

Of interest is the large amount of pottery found, including fragments of stirrup jars, deep and shallow cups as well as goblets, amphorae and alabaster (Sideris et al, 2017, 278). The pottery, based on typology and decoration, dates back to LH III B and C (Furumark, 1972). Regarding the subject of the work, 81 samples of ceramics were selected and analyzed with XRF and XRD methods, in order to identify the origin of the clay used for their construction.

These 81 specimens of fine and coarse pottery include handles, rims, bases and parts of the vessel bodies. In general, these are ceramic fragments that come from vessels of mainly closed type (thin) as well as from some pithos (coarse). All the fragments were carefully photographed, and some of them are selectively presented here (Fig. 4).



Figure4. Indicative pottery fragments under analysis

Specifically in Fig.4 are presented:

- K1: base of red slip ware
- K2: fragment with colored surface
- K67: decorated body part with handle trace
- K66: vessel mouthpiece
- K30: decorated ring-shaped base
- K16: handle (coarse)
- K62: upper part of stirrup jar (decorated with concentric circles)
- K48: rim vessel part

Some ceramic fragments have a characteristic decoration of the period, while others are not, or

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at least their decoration is not preserved. The ceramics were recorded one by one in excel boards with the code "K" (*cc Kastrouli*) and ascending number (K1-K81), together with their respective data (excavation data: barcode-locus-square-origin).







XRF analyzes were performed in the Laboratory of Archaeometry of the University of Peloponnese. Afterwards, the XRD analyzes were performed on the same pottery samples as well as on clay samples in the Laboratory of the Department of Geology of the University of Patras, under the supervision of Assistant Professor I. Iliopoulos. In the following chapters, explanations of the methods applied in the context of the work as well as the results of the respective analyzes are presented.

MATERIALS AND METHODS

Led Microscope – Characterization of Clays

Initially, in terms of methodology, a microscopic analysis was performed in the laboratory with the LED microscope on the surface of the samples in order to identify the clay and to make a first distinction between them according to the Munsell scale (color system). An optical microscope with a digital camera and a screen freezer function were used for more extensive observation of the object to be examined (Fig. 5). The clays were then identified according to the Munsell Color System scale (Munsell Soil Color Chart, 1992) in order to characterize the detected categories of clays.

Table1. Catalogue with Led microscope results

<i>Sample ID</i>	<i>Ceramic fragments</i>	<i>Clays by LED microscope</i>	<i>Munsell color diagram (values)</i>	<i>Munsell color chart</i>
K_60 <i>-rim-</i>			5YR – 5/6	Yellowish red
K_61 <i>-rim-</i>			2.5YR – 6/8 GREY2 – 6/1	Light red/ Bluish gray
K_62 <i>-upper part of stirrup jar-</i>			5YR – 5/8	Yellowish red

During the microscopic analysis, the surface of objects with the use of an electronic beam is examined, while at the same time with the use of a camera, the creation of an image of the examined surface on the computer screen is achieved.

So, the basic principle is to synchronize the beam scanning with the scanning of an external screen (TV or computer), while detecting the emissions produced by the essay and channeling them to the screen, where an image is created depending on the essay broadcasts (Goldstein J et al, 2003).







Figure5. Microscopic analysis

After photographing the clays of all the samples one by one, a detailed catalog was created containing photographs of all of them, their typology as well as the representation of each clay from the microscope (Table 1).

Therefore, twelve distinct categories occurred from the above process. Also notable are the resemblances among the samples (Fig. 6).

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<p>K_63 -handle with part of the body-</p>			<p>5YR – 6/6</p>	<p>Reddish yellow</p>
<p>K_64 -handle-</p>			<p>10YR – 7/4</p>	<p>Very pale brown</p>

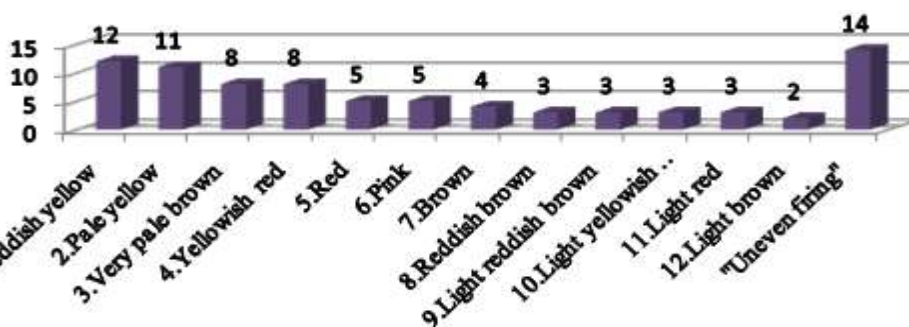


Figure6. Percentage ratio of 81 ceramic samples among the various clays

From the above distribution, it is concluded that items that had a common excavation origin (ie collected from the same area) belong to the same category of clay. This suggests that the pottery of a particular location (locus) was made with the same clay by the potters. Apparently, each potter used a specific type of clay and he also had the knowledge to create the pots he wanted. Clearly, this is not a coincidence, but an extracted information that was theoretically expected but is now being confirmed in practice.

Pottery Analysis by XRF Method

The X-ray fluorescence method is a total sample analysis technique. The depth of the analyzed sample ranges from less than 1 mm to 1 cm depending on the energy of the X-rays emitted and the composition of the sample (D.A. Skoog, F.J. Holler & T.A. Nieman, 2002).

An advantage of the method is also the ability to simultaneously analyze both elements those of high concentrations (100%) and trace elements in the area of ppm (Janssens, 2000). More generally, its importance lies in the fact that XRF spectroscopy is widely used for qualitative and quantitative elemental analysis of environmental, geological, biological, industrial and other samples.

Portability is a major advantage in sample analysis and makes it easier for scientists to provide secure results to the user. Portable devices of various

manufacturers and functions that apply to ceramics, clay and various materials are on the market. The choice of detector depends on the composition of the material under consideration (Zarkadas & Karydas, 2004).

The data collected from the analyzes with the XRF method, after being quantified, were included in separate tables for the separation of the results. With the application of two different operating conditions, as mentioned above, the oxides of elements and the heaviest elements contained in the 22 samples were detected.

In particular, it is the detection of the percentages of the oxides of Sodium, Magnesium, Argile, Pyrite, Potassium, Calcium, Titanium and Iron. One of the main advantages of white paints is the chemical inertia and the very fine grains for high quality paint (Tsirambidis, 2005).

The analyzes in the laboratory were performed with a portable XRF detector and specifically with Bruker's III-SD model. The areas of the samples analyzed were clean surfaces without coating (no preparation of the sample is required). For both XRF and XRD analyzes, 22 samples were selected of the total 81 (Fig.7).

These actions lead to groupings of samples and clear conclusions, which can be contrasted with the corresponding results of XRD.

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Through these results, the chemical elements of the selected pottery were identified (i.e. MgO, SiO₂). By combining them with the following XRD results, groups will occur that lead to the common clay used at the site.

The X-ray fluorescence method is a non-destructive method for the analysis of solid and liquid bodies. The sample is irradiated with a beam of X-rays that causes the emission of

"secondary" X-rays that are characterized as fluorescent. X-rays emitted can be detected by energy dispersion detectors (EDS) or wavelength dispersion (WDS).

The energy or wavelengths of the X-rays emitted are used to identify the chemical elements present on the sample under analysis while the data concentrations are determined by the intensity of the X-rays.

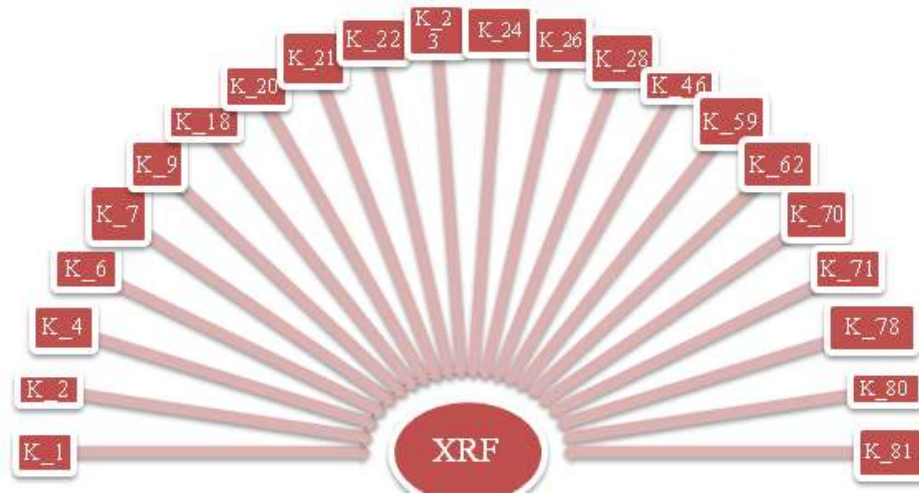


Figure7. Graph with the 22 ceramic samples analyzed by XRF and XRD

Cluster Analysis – Diagrams

Diagrams along with the chemical elements as variables are usually used after the analyzes (Liritzis, 2008, 433-450) in order to group samples in relation to the percentages of the detected data.

The following binary diagram shows the correlation

between the content of samples in Sr and TiO₂ while the creation of distinct ceramic groups is noteworthy for the study (Fig. 8).

In general, the variables Sr and TiO₂ can provide data on the origin of the clays, as long as the data are correlated with the wider geological environment of the site.

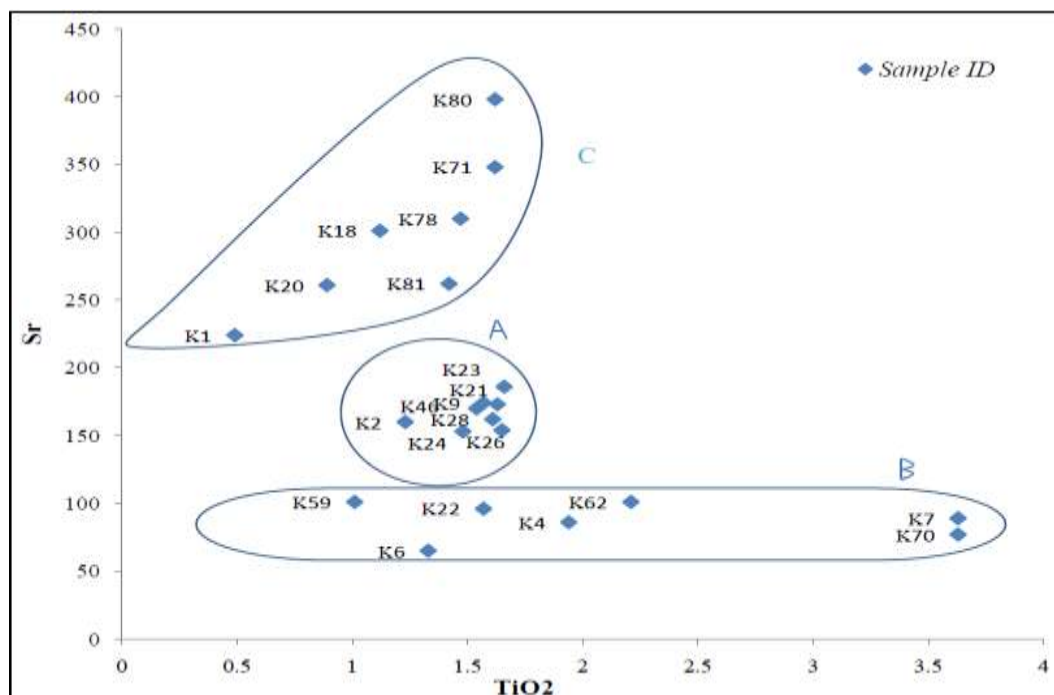


Figure8. Variable diagram of TiO₂ - Sr

Pottery and Soil Samples under XRD Analysis

The X-ray diffraction method was performed on the same above ceramic samples as well as on four soil samples in physical condition and after their experimental heating at 700°C and 900°C. The representative soil samples were collected from neighboring to the site areas in order to provide information about the origin of the clays (Fig. 9, DS=Desfina village).

The aims of using this analytical method were:

- The determination of the mineralogical composition of the samples under study and their characterization,
- The estimation of the firing temperature of the ceramics and
- The behavior of soil materials in their experimental heating at temperatures close to those estimated for the ceramic material under study.

Soil samples	Location
DS 1	Agia EIrini 1
DS 2	Agia EIrini 2
DS 3	Limnos
DS 4	Meteles

Figure9. Catalogue of soil samples under analysis

After the samples were pulverized, they were placed in tubes, where the amount collected (~ 2gm of dust) was measured, with the corresponding code of the sample (Figs 10, 11).



Figure10. Pulvering process at the Laboratory



Figure11. Placement in special tubes for the XRD analysis

Clay Brickets. Creation and Firing at the Laboratory

The creation of brickets from raw materials and their firing have been applied for years (Kreimeyer, 1987, 175-183), aiming at the identification of the factors that affect the sample under firing conditions and its differentiation at the end of the process (Fig. 12).

In the course of the study, it was deemed appropriate to study some of the technological properties of the soil materials collected from the study area regarding their suitability for use in the manufacture of ceramics; after the soil samples were baked as other ceramics, they were analyzed again with the XRD method.

Once the samples have been passed through the firing process, the results of the XRD will be able to be correlated with the results of the site's ceramic samples for the mineralogical composition of the clays (Fig. 13).

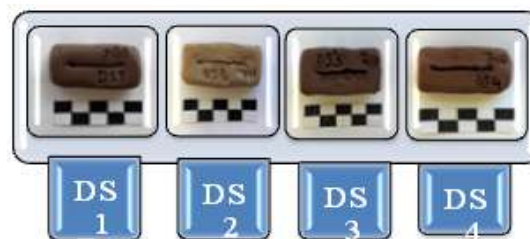


Figure12. Clay Brickets before their firing



Figure13. Clay brickets (DS2-DS3-DS4) after their firing at 900° C

Concluding with regard to clay brickets, two categories of clays appear that present mainly different characteristics. Their only similarity is in the loss of volume of all samples, which is a natural consequence of the reaction of the clays.

The following graph (Table 2) presents the properties of the clays observed during the creation of the brickets and their firing.

The non-calcium class is characterized by the limited presence of Ca-rich mineral phases consisting mainly of quartz, illite, potassium star, kaolinite and small amounts of calcite and aragonite (DS3-DS4).

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The calcium category consists of minerals: calcite, quartz, chlorite, oblique, while small amounts of dolomite, potassium and hematite were also identified (DS1-DS2).

The baked bricks were studied and categorized exactly like the pottery fragments as shown in Table 2 so that the results can be compared to each other.

Table 2. Variations of experimental bricks before and after their firing at temperatures of 7000C and 900⁰C

SAMPLE ID:		DS_1	DS_2	DS_3	DS_4
Munsell color	Wet	Brown (7.5YR 4/3)	Pale brown (10YR 6/3)	Brown (7.5YR 4/3)	Brown (7.5YR 4/3)
	Dry	Light brown (7.5YR 6/3)	Very pale brown (10YR 8/2)	Brown (7.5YR 4/4)	Brown (7.5YR 4/4)
	Fired (700 ^o C)	Light reddish brown (5YR 6/4)	Pink (5YR 7/4)	Red (2.5YR 5/8)	Red (2.5YR 4/8)
	Fired (900 ^o C)	–	Light gray (10YR 7/2)	Red (2.5YR 5/8)	Red (2.5YR 5/8)
Bar measurement (shrinkage)	Wet	3	3	3	3
	Dry	2.7	2.7	2.6	2.6
	Fired	2.9	2.9	2.6	2.6
Bar weight (porosity)	Wet	25.34	21.69	24.07	20.75
	Dry	20.62	16.64	18.45	16.62
	Fired	16.55	13.02	15.49	14.26

Determination of the Mineralogical Composition of Ceramic Samples

The mineralogical components identified in all the analyzed samples as well as the semi-quantitative estimate of the participation of each of them were studied. Based on the mineralogical recommendation that emerged, almost all the ceramic samples analyzed except one (sample K78), seem to predominate in calcium, i.e. with a CaO rate greater than 6% by weight.

A total of three main sample classes emerged depending on the general data observed, A, B and C. The classes are described below in parallel with the corresponding results obtained from XRF and XRD analyzes.

DISCUSSION

Characterization of Ceramics

In conclusion, the pottery under consideration is characterized as fine-grained in terms of quality and of calcium in terms of mineralogy. The categorization of the ceramic samples leads to the convergence of the results on both sides.

It is worth noting that there is a significant correlation with the mineralogical data and the three classes determined on the basis of these data in relation to the estimated firing temperature of the analyzed ceramic samples (Fig. 14).

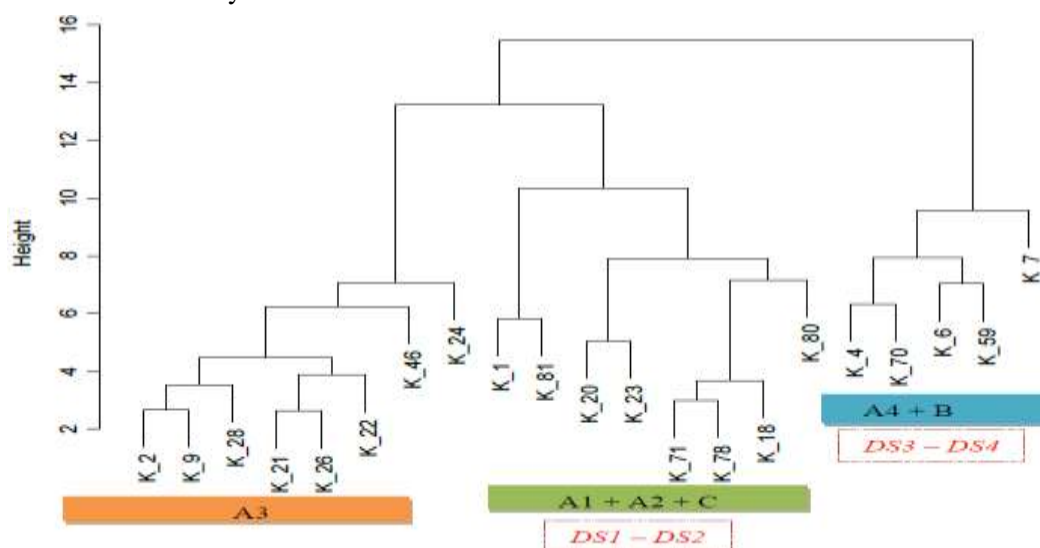


Figure 14. Correlation of the constituent groups derived from the statistical processing of chemical data (XRF) with XRD rated temperature classification (orange color: group I, green color: group II, blue color: group III)

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Specifically, group I includes all samples of radiographic class A3, group II includes almost all two classes A1 and A2 (with the exception of sample K22), while group III includes half of the samples of group A4 (except K28 and K62) and almost all samples of group B (except sample K2).




From the samples that do not follow the above general categorization scheme, sample K2 is included in group III, sample K22 in group I while sample K62 was not included in the statistical processing for the reasons mentioned above.

The above statistics (Fig. 14) confirm the correlation of the data through the convergent groupings and therefore indicate with certainty the identification of three different structural categories of ceramics.

To understand the results in relation to the ceramics examined, three tables follow with the three general categories extracted on the basis of chemical and mineralogical analyzes (Tables 3-5). The tables contain the illustrations of the pottery per group / class that were found in relation to the raw materials, so that the results can be understood visually too.

Specifically, Table 3 shows the 8 fragments of the first category, Table 4 lists the other 8 samples of the second category, and finally, Table 5 shows the 5 items of the third and last category.

Table3. Correlation and identification of chemical and mineralogical data by depicting the eight ceramic samples included in class A3 (XRD) and group I (XRF)

Sample ID	Ceramic fragments	Data correlation (XRD/XRF)
K_2		A3 / Group I
K_9		
K_21		










K_22	
K_24	
K_26	
K_28	
K_46	

Table4. Correlation of chemical and mineralogical data with the illustration of the eight ceramic samples included in classes A1+A2+C (XRD) and in group II (XRF)

Sample ID	Ceramic fragments	Data correlation (XRD/XRF)
K_1		A1+A2+C / Group II (DS1 - DS2)
K_18		
K_20		
K_23		










K_71	
K_78	
K_80	
K_81	

Table 5. Correlation and identification of chemical and mineralogical data with the illustration of the five ceramic samples included in classes A4 + B (XRD) and group III (XRF) - Correlation of raw materials

Sample ID	Ceramic fragments	Data correlation (XRD/XRF)
K_4		A4+B / Group III (DS3 – DS4)
K_6		
K_7		
K_59		
K_70		

Origin of Raw Materials

Regarding the issue of the origin of the ceramics under consideration, the chemical and mineralogical data of the ceramics can be correlated with the corresponding ones of the raw materials. The final groupings of the samples obtained from the re-statistical processing of the data, showed a small change as the sample K_6 (Table 5) was included in the second category of samples, as can be seen in Fig.81.

Chemical analysis of the raw materials and ceramics led to the conclusion that the two soil samples DS1-DS2 were identified with the category presented in Table 4 and now includes 9 samples of ceramics. While, the DS3-DS4 samples coincided with the third category presented in Table 5 and includes 4 ceramic samples.

From a mineralogical point of view, the mostly calcium ceramics of the site under study appear to be related to the DS1-DS2 calcium samples from the area of Agia Irini. The technological properties of these raw materials that were recognized before and after they were baked converge with the characterization of the ceramic samples.

The graph in Fig. 14 shows the final grouping that resulted from the statistical processing of all the data, including the ceramic samples and the raw materials. This grouping confirms the conclusions mentioned above regarding the three general categories identified between the samples.

The predominant category (Table 3) includes most of the fine ceramic specimens that coincide with the DS1-2 calcium bricks. The estimated firing temperatures of this class range, in general, from 800°C - 1000°C.

It is worth noting that most pottery fragments, the clay of which was identified in the raw materials from Agia Irini, include vessel bases and body parts mainly of fine light-colored pottery (Table 4) belonging to closed vessels such as decorated stirrup jars.

As for the samples, the clay of which was identified with the soil samples from the areas of Limnos and Meteles (Table 5), these are coarse shells of red clay with the parallel addition of inclusions. Besides, it is generally known that ceramics were preferred during the Mycenaean period in light-colored clays, which were mainly used for the construction of written pottery.

The question of the origin of the 8 ceramic samples of the chemical group I / class A3 remained unanswered, as these fragments do not

coincide with the examined raw materials. Probably, the clay of these samples comes from another area, since the chemical and mineralogical analyzes did not indicate any kind of correlation between them.

Therefore, with regard to the raw materials examined, the data support the basic origin of the raw materials for the construction of the ceramics examined, from Agia Eirini, from where the soil samples DS1-DS2 that prevailed in total were collected. At the same time, it appeared that a small part of the pottery under consideration coincides with the raw materials from Limnos and Meteles. The process of burning the vessels is estimated to have taken place under the corresponding recommended temperatures proposed above according to the mineralogical analyzes of the ceramic specimens.

Thus, we are led to the hypothesis that the potters of the Mycenaean site could obtain their clay mainly from the neighboring area of Agia Irini and less from the other areas. This possibility of the origin of the materials seems to be justified by the fact that "Kastrouli" is adjacent to Agia Irini and the transport of raw materials would be an easy task. In addition, the preference for clay from Agia Irini probably indicates the quality of these raw materials for the construction of its ceramics.

Therefore, it is noteworthy that the examination of the materials revealed the use of local materials (soil/clay) for the construction of pottery, which is a key feature of a settlement as it is directly related to the daily activities of the inhabitants (useful vessels) but also to their votive habits (as gifts to the graves).

It is recalled that the results concern a part of material that came to light from the first excavation period of the site during the summer of 2016, which focused on Tomb A and two trenches in its wider area.

The conclusions for the pottery under consideration were drawn up in relation to the indicative raw materials that were also examined.

Certainly, the forthcoming systematic excavation activities in the Mycenaean site will bring even more material to light, highlighting its character and importance. Clearly, further study and analyzes of the findings in the future will answer the questions that arise around the Prehistoric position in greater extent. "Kastrouli" is an important place that came to complete the Mycenaean map of Greece.

ACKNOWLEDGEMENTS

The present master thesis was developed in the framework of the Postgraduate Studies Program "Applied Archaeological Sciences" in the Department of Mediterranean Studies of the University of the Aegean (Rhodes, 2017).

I would like to thank Prof. N. Zacharias and Prof. I. Iliopoulos for their participation in the present study with the availability of their University Laboratories at Kalamata and Patras respectively, as well as for their significant advice.

At the same time, I thank the PhD candidates Eleni Palamara and Vagia Xanthopoulou for their help at the Labs regarding the implementation of the analyzes.

Finally, I thank Prof. A. Sampson for his accurate comments on the writing of the text.

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Citation: Tonia Tsourouni, "The Case of the Mycenaean Site of 'Kastrouli' Near Delphi; Characterization of Pottery and Clay Material: A First Assessment of the Results through XRF and XRD Analyses", *Annals of Archaeology*, 3(1), 2020, pp. 30-40.

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