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DIPLOMA THESIS:

SEM-EDS analysis of mortar samples collected from the large jar complex at Kolona, Aegina for the identification of their use

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SEM-EDS analysis of mortar samples collected from the large jar complex at Kolona, Aegina for the identification of their use

George A. Vlahos

Abstract

The present thesis focuses on the analysis of seven unique mortar samples, collected from the area of three large jars excavated at the island of Aegina, Greece on September 2020 at a location known as "Paralia" few kilometers north from the archaeological site of "Kolona" inside the walls of the ancient city. More specifically the exact shape of the large jars, which were built one next to the other, highly resembles that of open tubs. The microstructure, the chemical composition and the effects of the presence of sea salt on the samples were studied through the microscopic and chemical analysis. The analyses have mainly resulted in the chemical composition of the mortar that was covering the interior of the large jars, whereas archaeological insight derived from the archaeological excavation in the large jar area at "Paralia" as well as the detection of sulfur trioxide (SO_3) during the analysis with SEM-EDS. These were the key factors that allowed the correlation of the large jars use with the fulling process (i.e. clothes/textiles washing and refining) and thus the characterization of the whole establishment - wherein the large jars were found - as a fulling workshop.

Introduction

The archaeological site of Kolona is one of the most important sites on the island of Aegina that encompasses the ancient history of the area from the Early Bronze Age until the Late Roman/Early Byzantine period (3300 BC - 9thc. AD). Among the remains of several dwellings, the ruins of the temple of Apollo constitute the most prominent structure inside the area that is known as the acropolis of the ancient city of Aegina (Jennings, 1988). Just a few kilometers north from the acropolis the remnants of three walls encircling three large jars were excavated on September 2020 at an area named "Paralia", as indicated on the map below (fig. 1). However, from this point on the name "Kolona" will be used to signify the location of the large jars, as they are located at a short distance from the ancient settlement of Kolona. The discovery of the site was based on the remains of two walls - wall 1 (north) and wall 2 (south) - that were still visible at the time when the excavations started. These walls were extended towards the coast, while scarce stones from these walls were found under the sea water. These walls actually mark the perimeter of the excavated area - 10x5 in dimensions - both at its north and south sides respectively. Apart from the walls, parts of the exterior surface of two ceramic vessels were also visible prior to the excavation. During the archaeological survey three large jars in the form open tubs (fig. 2) as well as a third wall 3 (eastern) behind them that constitutes the eastern side of the excavated area came into light (fig. 2). In particular, the lower part of large jars was integrated in the establishment in order three large open tubs as shown in fig. 2 to be formed (Kalamara P., 2020). Several building materials (e.g. stone) have been used to give a better form to the upper part of each large open tub (Kalamara P., 2020). From this point and the large open tubs will be referred to as large jars.

Fig. 1 Left: The map of the island of Aegina. The exact location of the large jars area is indicated by the red pointer. Right: A general view of the excavated site from the south side is depicted on the right photo (© Ministry of Culture EEA / Aix-Marseille Université – CCJ – MoMArch / EFA, φωτογραφία © P. Kalamara 2020). The north, south and eastern walls are pointed out by the red, blue and green pointers respectively.

Fig. 2 Left: The large jar 3 interior that was recovered in fragmentary form is shown after the end of the excavation. South of that large jar the artificial walls ("narthex") that was used to support the detached west side of the large jar 2 is also indicated by the red pointer (© Ministry of Culture EEA / Aix-Marseille Université – CCJ – MoMArch / EFA, φωτογραφία © P. Kalamara 2020). Right: The three large jars 1, 2, 3 as shown from above after the end of the excavation (© Ministry of Culture EEA / Aix-Marseille Université – CCJ – MoMArch / EFA, φωτογραφία © P. Kalamara 2020).

The large jars interior was covered by white mortar, which was used in order to create a smooth surface as well as to shape the rim of the jars unifying them in an array of three ceramic vessels in the N-S direction. The thickness of the mortar was varying as it was thicker (around 2 mm) on the upper part of the large jars inner surface and less thicker toward its lower part, where the mortar was not macroscopically visible. According to the archaeological data the variation in the mortar thickness has resulted from the technique used for the large jars construction rather than from the effects of corrosion (Kalamara P., 2020). The same coating material was used on the area around the large jars as well as on the surface of the eastern wall. No traces of that mortar were found in the large jar 3 interior, because it remained uncovered for a long time period prior to the excavation making it impossible (i.e. mortar) to survive nowadays. In addition, broken roof tiles were found spread all over the large jars complex in contact with the wall 3. These tiles could possibly be the remnants of the roof that was covering the whole establishment, wherein the large jars have been constructed, although this has not yet been clarified with certainty. In general, the archaeological material connected with the establishment that was once standing on that place is highly fragmentary and it can be dated to the Late Roman (284-476 AD) and Early Byzantine period (323-717 AD). The basic research question was about the composition of the white mortar that was covering the large jars interior and the area around them. So, the current research aims at the elucidation of the white mortar properties as well as the identification of any residues that may have remained on its surface and would reveal information about the use of the large jars.

Samples and methodology

In this section the sampling process as well as the methodology that was followed during the research will be presented in detail (fig. 3). In total seven mortar samples were collected from the large jars complex that was found at Kolona as shown in table 1. As we can see each mortar sample is composed of several mortar fragments that were documented by taking multiple photos of each mortar sample with the use of camera in order to obtain a satisfactory number of pictures in the research archive (see table 2).In the second phase (Optical microscopy) each mortar fragment from each sample was examined under the optical (Leica Zoom 2000) and LED microscope (I-Scope Moritex) in order to study their visual characteristics and more specifically the color, any pores or visible inclusions on the samples surface and the situation, at which the mortar samples were collected in general.

Table 1 The table presents all mortar samples collected from the large jars complex, the number of mortar shards of each sample as well as the exact spot, where each sample was collected from prior to the examination with OM and LED microscope and SEM-EDS.

#	Name	No of mortar fragments	Collection spot
1	**C**	2	Contact spot of the large jar 1 with the south wall

2	**WD**	5	Contact spot of the large jar 1 with the south wall
3	**Large jar 1**	5	Large jar 1 interior
4	**P1**	15	Large jar 1 interior
5	**P3**	2	Area between eastern wall and large jar 3
6	**Powder 1**	In powder form	Large jar 1 interior (high)
7	**Powder 2**	In powder form	Large jar 1 interior (low)

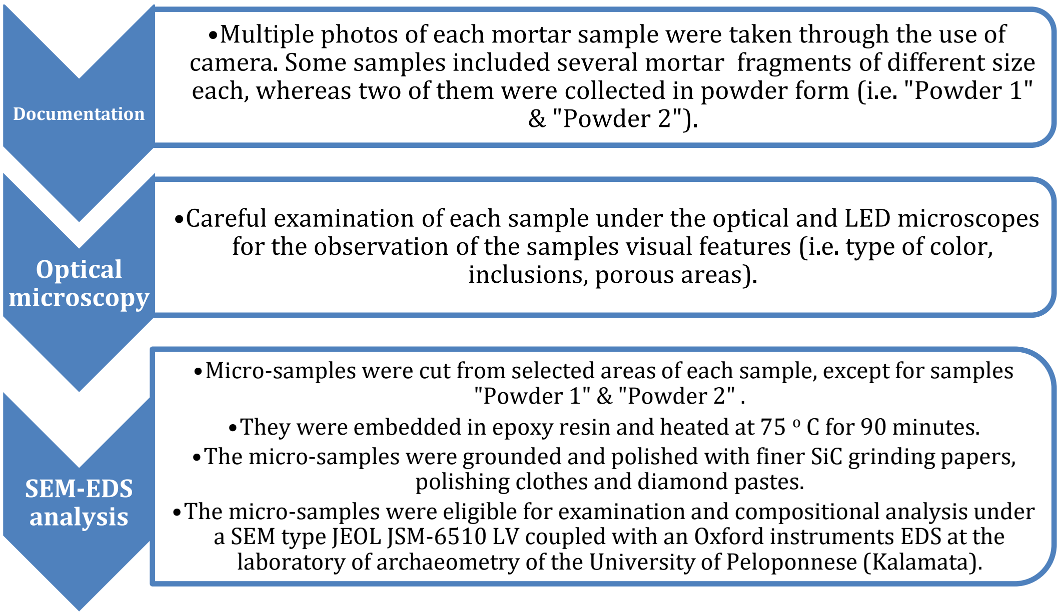
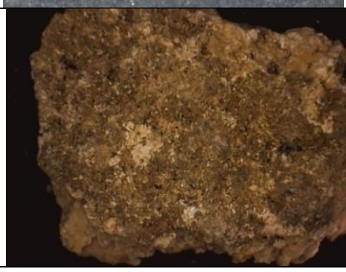
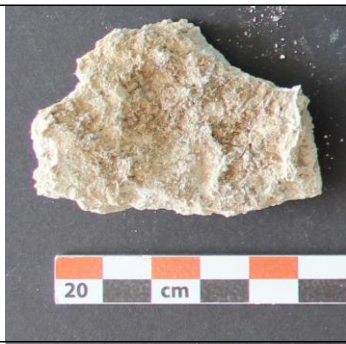


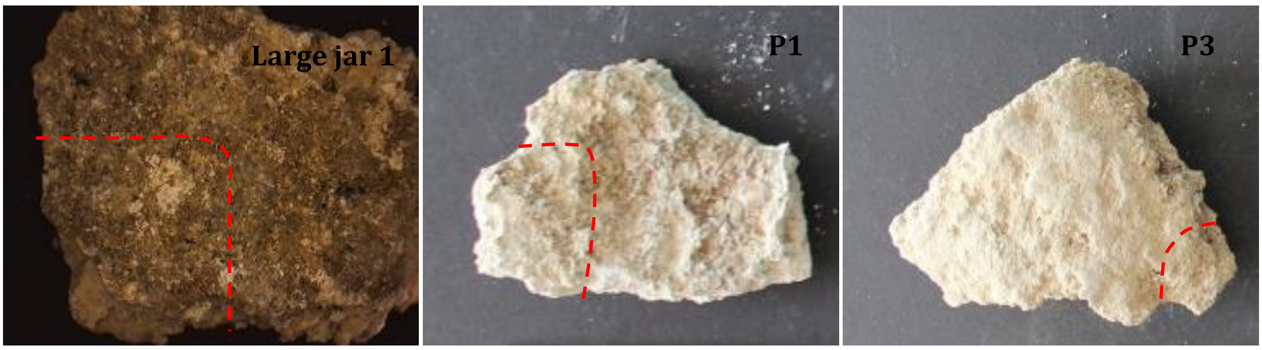
Fig. 3 All steps of the methodology followed (i.e. documentation, optical microscopy, SEM-EDS analysis) during the research are presented in the figure above.

In order to conduct the SEM-EDS analysis, micro-samples were cut off from selected areas of each mortar sample (except for “Powder 1” & “Powder 2”) using a handheld rotary tool (Dremel) with a saw blade attached to it. The samples that were chosen for the analysis with SEM-EDS are presented in table 2 as well as the photos below showing the exact micro-sample that was cut from each mortar sample.

Table 2 The micro-samples that were analyzed with SEM-EDS are shown in the table below. Samples C1 & C2 derive from the sample C shown in table 1, whereas samples WD1 & WD2 have come from the sample WD.

Sample	**Micro-sample**	**Photos**
C	C1	
C	C2	
WD	WD1	
WD	WD2	
Large jar 1	Large Jar 1	

P1	-		
P3	-		
Powder 1	Grains 1-5		
Powder 2	Grains 1 & 2		



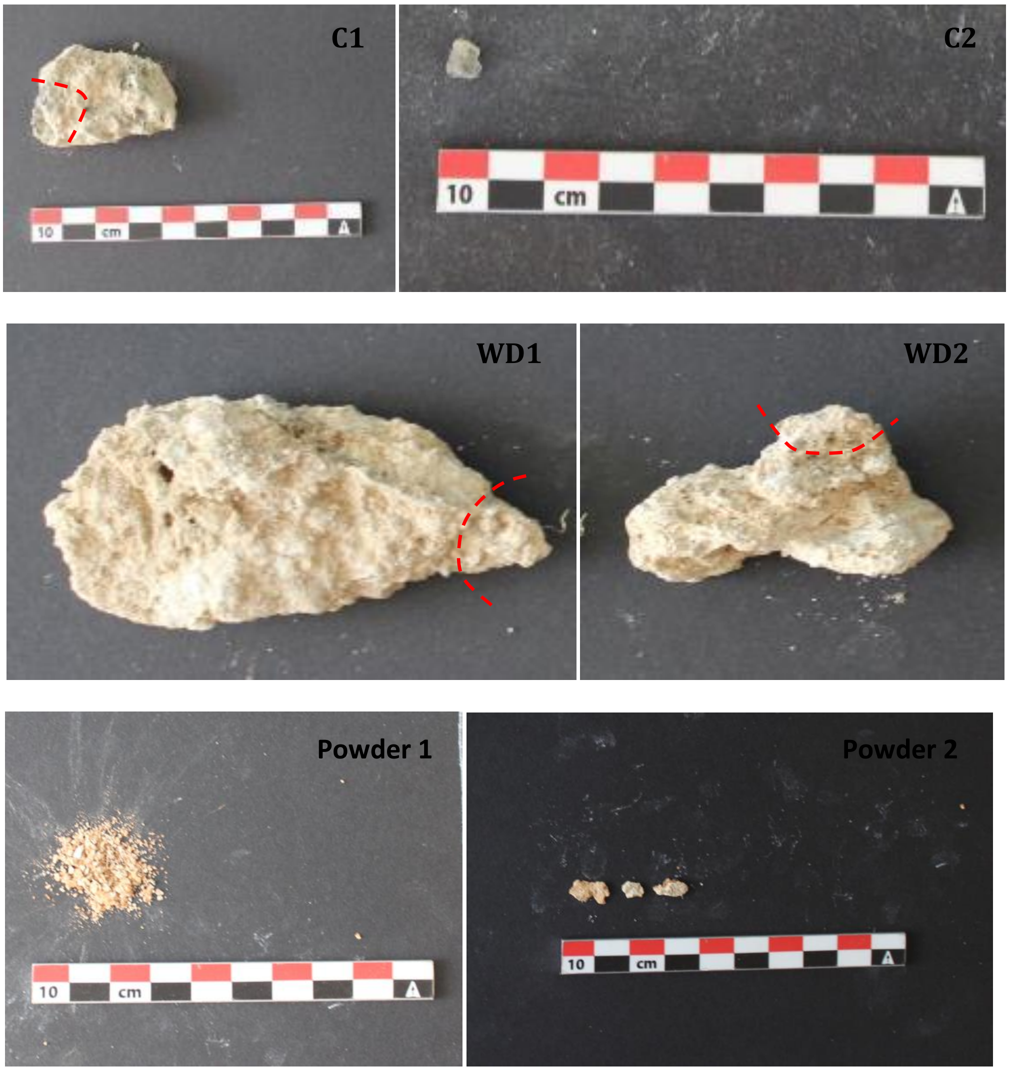


Fig 4 The micro-samples that were selected for the analysis with SEM-EDS are presented in the pictures above. The red dashing lines have been used to point out the exact micro-sample cut from each one of these fragments. The largest grains from the samples “Powder 1” and “Powder 2” were chosen for the analysis of these two samples.

The micro-samples were embedded in epoxy resin (CaldoFix-2 kit) and heated at 75°C for one hour and thirty minutes. They were then grounded down with progressively finer silicon carbide grinding papers (grit 180, 320, 400, 800, 1200, and 2000) and then polished with a series of polishing cloths (6 μm, 3 μm, 1 μm, 0.2 μm) and diamond pastes. Grinding and polishing was carried out using a Labopol-2 device by Struers. The polished samples were analyzed under a SEM (Scanning Electron Microscope) type JEOL JSM-6510LV coupled with an Oxford Instruments EDS (Energy Dispersive Spectrometer). The analytical data were obtained by INKA software. All analyses were conducted at high vacuum, 20 kV accelerating voltage and with a count time of 120 sec(Moropoulou et al., 2018). Bulk and point analyses as well as mapping of the major chemical compounds contained in the samples were conducted at a magnification of x100. Each sample was measured at least three times(Moropoulou et al., 2018).

Results

i)Examination with optical and LED microscope

The examination of the mortar samples under the optical and LED microscopes was a necessary procedure before we analyze the samples with SEM-EDS. In particular, all fragments of each mortar sample were examined carefully aiming at the detailed documentation of the characteristic features of their surface. Regarding the color of the mortars the Munsel soil color chart 1994 revised edition was used in order to define the different hues of each sample. Brown is the color of the vast majority of the samples with "very pale brown 10YR, 8/2" to be the most frequent hue that was identified in mortar fragments. "Reddish brown 2.5, 5/4" was spotted only twice (fig. 5) in some grains of the two samples collected in powder from ("Powder 1" & "Powder 2"), whereas a completely different hue - "light yellowish brown 10YR, 6/2" - has been identified once on the one side of the sample "P3" (pale brown is the color of the other side). These are the basic colors that were observed on the mortar samples, although other colors have been occasionally recognized too. For instance, green color and more specifically "greenish black GLEY, 10YR, 2.5/1" is visible on the surface of the sample "C" as a result of the oxidized copper found on that sample (fig. 6). Moreover, "light brownish grey 2.5Y, 6/2" is the color of a specific area on the sample "C", on which pores of varying sizes appear (the sole porous area found on any of the mortar samples) that seem to have derived from the development of high temperatures as it will be further discussed later on (fig. 6). No other porous areas have been found in the rest of the mortar samples apart from some crevices that render the samples quite brittle. Finally some white - colored areas "10YR, 8/1 probably rich in calcium oxide (CaO) have also been reported sporadically in the samples "P1", "P3" and "WD".

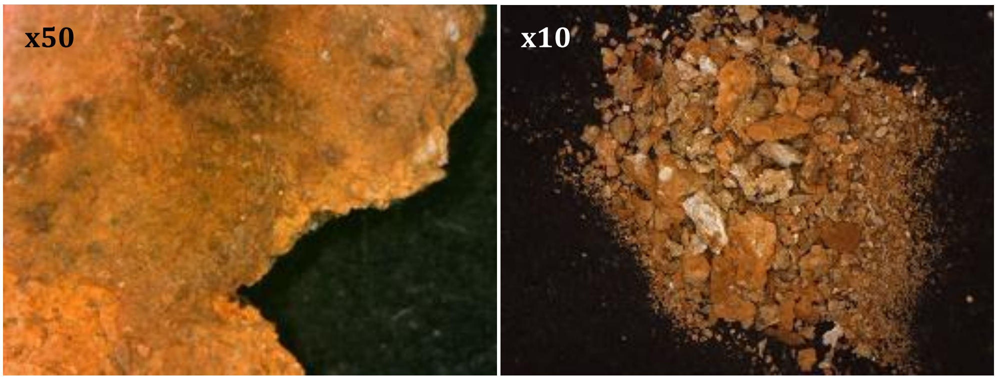


Fig. 5 The LED image above was taken at a very short distance from one of the particles of the sample "Powder 1" showing the reddish brown color of its surface. The image on the right depicts the sample "Powder 2" and especially the extremely small particles that form part of that sample.

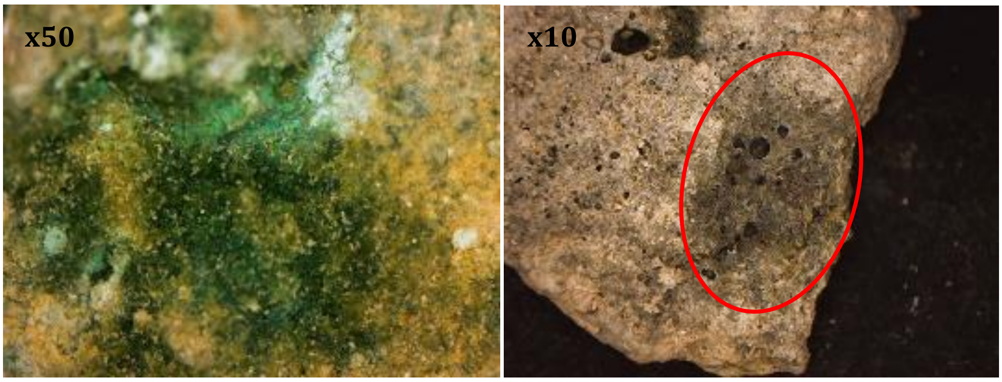


Fig. 6 Two characteristic areas of the sample "C" are shown in the pictures above. The impact of the oxidized copper can be clearly seen on the green areas, while the only porous area found during the examination of the mortar samples is marked on the right picture (red circle).

Several white spots or more extended white - colored areas were also brought into light during the samples OM and LED microscope investigation that should be characterized as purer areas of the white mortar, such as the white surface from the sample "P3" (fig. 7), while others have been traced in the form of rounded spots resembling residues of sea salt. We should keep in our mind the fact that the large jars have been excavated at a very short distance (just few meters) from the coastline of the island of Aegina, thus justifying a possible presence of salt on the mortar that was coated on the large jars. Areas of purer white mortar similar to that found in "P3" were also detected on the sample "WD" as shown in fig. 8. The surface of the samples is uneven in most cases except for the sample "P3" and particularly one of its sides that is quite even and smooth with very few aggregates to be visible. More specifically some inclusions have been spotted on each sample with varied color. Black, white and brown are the most frequent identified colors of the aggregates, whereas grey, purple, red and blue are rarer cases mostly found at the sample "C" suggesting that their color could be affected by the presence of the oxidized copper. Lastly, organic material in the form of roots, which was not related to the mortar material, was integrated in the mortars surface, the presence of which should be attributed to the fact that the large jars were buried under the ground for a long time, thus allowing the roots of the vegetation to grow deeply into the ground exactly above the excavation site. The most significant information on the mortar samples visual characteristics is briefly presented in table 3.

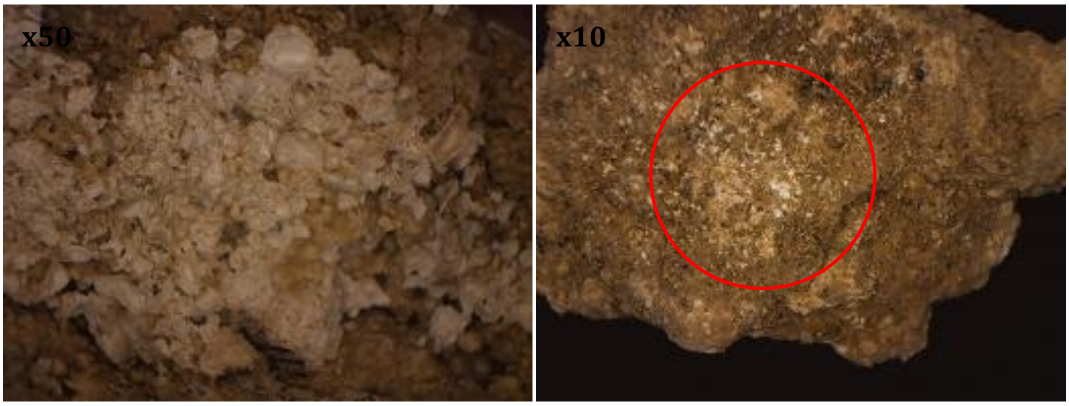


Fig. 7 Two areas of the surface of “P3” and “Large jar 1” respectively are depicted in the photos above. On the left picture part of the white mortar surface was found on the sample “P3”, whereas sea salt traces are shown as white spots on the surface of the sample “Large jar 1”.

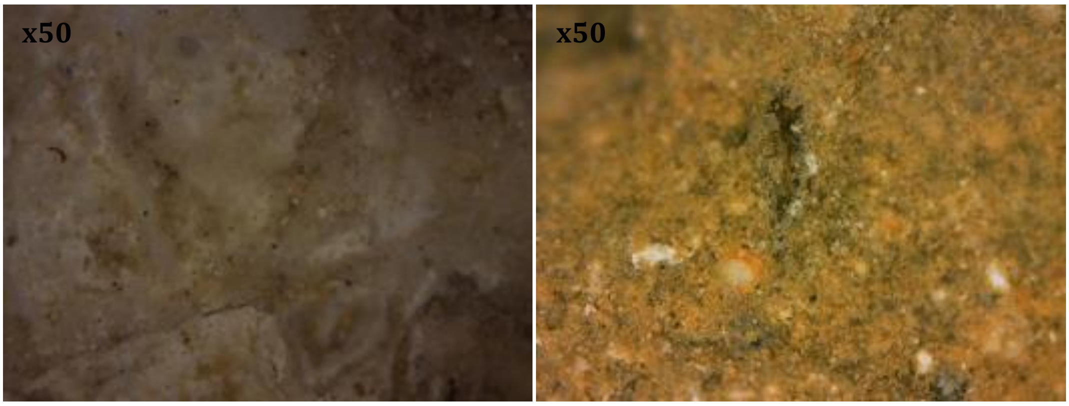


Fig. 8 Another surface of pure mortar is illustrated on the left LED image of one of the mortar fragments of the sample “WD” (“WD1”. The right LED image was taken from another fragment of the same mortar sample, where several aggregates of various color and shape have been found at.

Table 3 The table below summarizes the visual characteristics of the mortar samples as recorded through the OM & LED microscope use.

Sample	Color	Porosity	Aggregates
C	Pale brown	Yes	Very small rounded white and purple aggregates
WD	Pale brown	No	Strong variability in the color (black, white, brown, purple, red, blue, grey)
Large jar 1	Pale brown	No	Scarce aggregates of black, white and brown color
P1	Pale brown	No	Black, brown and white aggregates
P3	Light brownish grey (side 1) & pale brown	No	Aggregates of various sizes, shape (rounded, angular)

			and color (black, brown, white & grey) have been recorded
Powder 1	Reddish brown	No	Very few and small aggregates of white and grey color
Powder 2	Reddish brown	No	Very few and small aggregates of white and grey color

ii) SEM-EDS analysis

iia) Mortar microstructure and chemical composition

The analysis of the mortar micro-samples (fig. 4) with SEM-EDS produced a clear picture upon their microstructure and chemical composition. The average value and the standard deviation of multiple analyses for each micro-sample are presented on table 4. As we can see in table 4 calcium oxide (CaO) and silicon dioxide (SiO_2) are the two major components of the mortar suggesting that the mortar coated on the large jars interior as well as their surrounding area is a calcium-based mortar mixed with Si-rich aggregates. The relatively low concentration of both magnesium and sulfur excludes the use of dolomitic limestone and gypsum, respectively. Therefore, the mortar in all cases is made of calcareous limestone.

Table 4 The chemical composition of each mortar micro-sample is presented in the table above. The rates of each chemical compound is expressed on average (M.V.: Mean Value) along with the standard deviation (S.D.).

Sample		**Na_2O**	**MgO**	**Al_2O_3**	**SiO_2**	**SO_3**	**Cl^-**	**K_2O**	**CaO**	**TiO_2**	**Fe_2O_3**	**CuO**	**SnO_2**	**Total**
C1	M.V.	3.65	4.42	10.75	53.53	1.08	1.02	1.31	11.3	-	4.56	4.56	5.2	100
	S.D.	0.34	0.25	0.87	0.23	-	0.16	0.29	0.7	-	0.4	0.4	-	
C2	M.V.	2.51	5.19	10.97	53.14	-	0.86	1.82	16.4	0.99	5.97	2.28	-	100
	S.D.	0.21	0.08	0.79	1.25	-	0.09	0.4	0.6	-	0.56	0.45	-	
WD1	M.V.	3.95	4.49	1.46	49.54	3.41	5.37	1.15	27.51	-	3.95	-	-	100
	S.D.	0.64	0.45	0.34	2.58	0.38	0.21	0.64	3.33	-	0.64	-	-	
WD2	M.V.	3.59	3.36	3.21	72.01	1.95	3.98	1.09	11.72	-	2.11	-	-	100
	S.D.	0.68	0.21	0.38	3.42	0.17	0.49	0.25	0.51	-	-	-	-	
Large jar 1	M.V.	3.92	2.91	3.92	14.26	2	1.68	1.29	68.28	-	2.05	-	-	100
	S.D.	1.03	0.65	1.03	1.4	0.35	0.82	0.66	2.13	-	0.26	-	-	
P1	M.V.	-	-	-	12.56	-	-	-	89.25	-	-	-	-	100
	S.D.	-	-	-	0.72	-	-	-	4.6	-	-	-	-	
P3	M.V.	-	-	-	6.99	-	-	-	93.02	-	-	-	-	100
	S.D.	-	-	-	0.76	-	-	-	0.76	-	-	-	-	
Powder 1	M.V.	6.32	4.03	4.27	9.46	2.79	9	0.39	61.62	-	1.7	-	-	100
	S.D.	0.84	0.69	0.72	0.45	0.94	0.97	0.14	3.83	-	0	-	-	

Powder 2	M.V.	2.39	3.77	5.61	11.61	2.14	2.38	0.82	71.28	-	-	-	-	100
	S.D.	1.03	1.14	1.57	2.18	0.91	0.23	0.49	4.98	-	-	-	-	

The analysis of the samples "Large jar 1", "P1", "P3", "Powder 1" and "Powder 2" have resulted in the highest CaO rates on average that have been detected ranging between 61.62 and 93.02 wt %. Apart from the sample "P3" (collected from the area between the large jar 3 and the eastern wall) all the aforementioned samples have been collected from the inner surface of the large jar 1 suggesting that the mortar that was coated in the upper part of its interior is a calcium-rich one that was probably used in order to enhance the interior walls durability and resistivity to water. In particular, the extremely high CaO content found in "P1" and "P3" (89.25 & 93.02 wt %) suggests the use of a very fine and good quality mortar. The SEM images in fig. 9 illustrate the microstructure of nearly the whole surface of the two micro-samples, where the lighter colored the surface is shown the more CaO content has been traced. The relatively lower CaO amount found in "Large jar 1", "Powder 1" and "Powder 2" (68.28, 61.62 & 71.28 wt %) in conjunction with the rest of the compounds detected (SiO_2, Al_2O_3, Fe_2O_3, MgO, K_2O) and especially with the increased SiO_2 compound (9.46-14.26 wt %) suggests larger Si-rich aggregate content mixed in these three mortar samples. Fig. 10 and 11 depict a clear picture about the microstructure of these three mortar samples. The dark - grey areas on the images correspond to areas of the samples surface, where more SiO_2 was found.

It is particularly interesting to note that the sample Powder 2 (extracted from the base of the large jar 1), belongs to this category of fine, good quality mortar. According to the report that summarized the outcome of the archaeological excavations conducted in September 2020 the mortar was mainly covering the upper part of the large jars inner surface and no traces of it were found on the rest of the surface raising a lot of concern whether that mortar had been coated on the entire inner surface of the large jars or not. The analysis of the two grains that form the micro-sample "Powder 2", wherein 71.28 wt % of CaO was detected on average, indicates that the mortar may have been coated on the entire inner surface of the large jar 1 (reaching until its base).

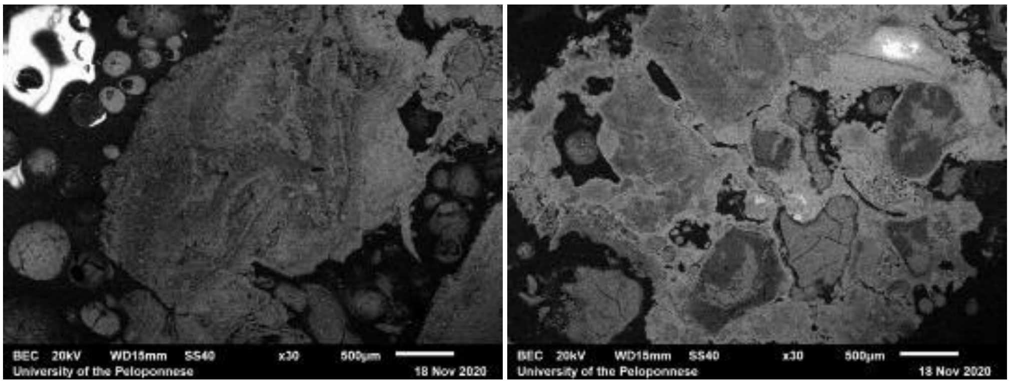


Fig. 9 The SEM images depict almost the entire analyzed surface of the "P1" (left) and "P3" (right) micro-samples.

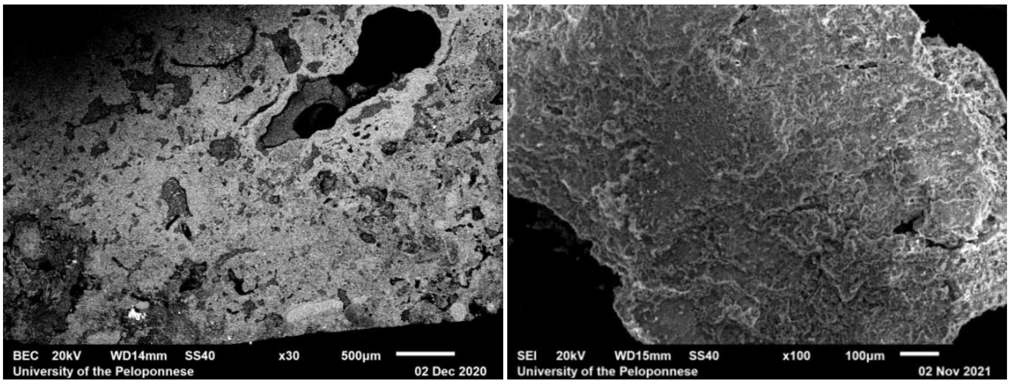


Fig. 10 SEM images of the micro-samples "Large jar 1" (left) and "Powder 1" (right).

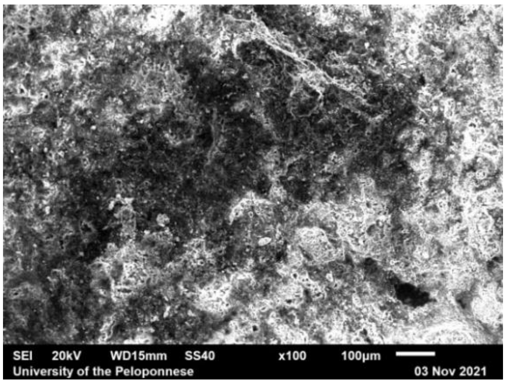


Fig. 11 SEM image of the sample "Powder 2".

On the other hand, the situation is quite different regarding the composition of "C1", "C2" (fig. 14), "WD1" (fig. 12) and "WD2" (fig. 13). There SiO_2 is present in far larger amounts when compared with the calcium-rich samples mentioned previously, spanning between 53.53 (C1) and 72.01 wt% (WD2). Thus, Si-rich aggregates are the dominant substance that is contained in these samples in comparison with the calcium-rich content. However, it is worth underlining that during the analysis of "WD1" and "WD2" several areas rich in CaO (77.4 wt% on average but not included in CaO rates in table 5) were also found (mostly in "WD1") suggesting that these micro-samples are a poor mixing of the calcium-rich mortar with the Si-rich aggregates. This can be mainly seen through the measured CaO in “WD1” (27.51 wt %), which is significantly larger than the amount of the same compound detected in the other three silicon-rich samples (“C1”, “C2” & “WD2”). In addition, the elemental mappings shown in fig. 12 (right) and 13 (right) show characteristic images of the samples, highlighting the poor mixing of the mortar samples WD1 and WD2 respectively. The red-colored areas have mapped the distribution of CaO content that was measured in extremely high levels, whereas the silicon-rich areas are illustrated with green color.

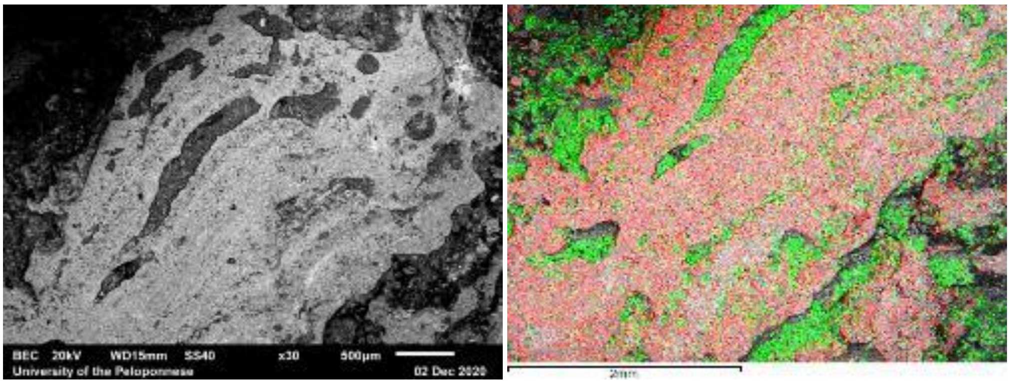


Fig. 12 The SEM images above have captured part of “WD1” surface that reveals the bad quality of the mortar. A characteristic calcium-rich area is shown on the right image, while the distribution of CaO (red) vs SiO_2 (green) is presented in the elemental mapping (left).

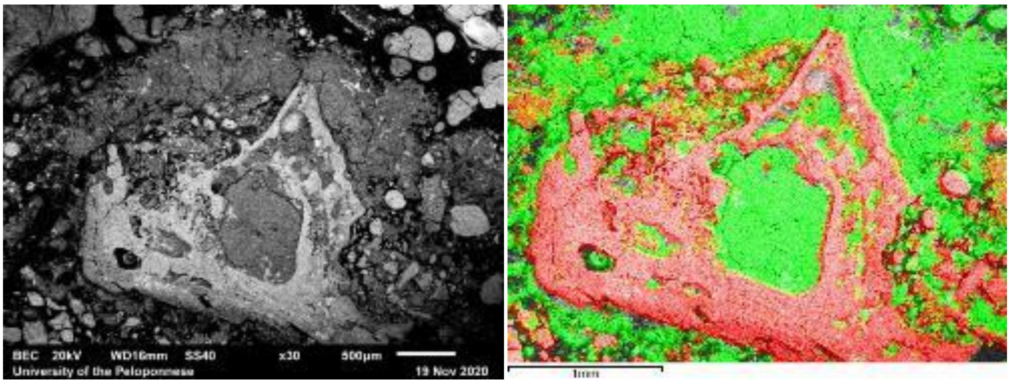


Fig. 13 Another area that shows the bad condition on the mortar found in “WD2” is depicted on the SEM images above. The white area on the left image is rich in CaO, the distribution of which is presented in the red-colored areas on the right image. SiO_2 has been traced in abundance in the green areas .

Sulfur trioxide (SO_3) has been traced in six out of the nine micro-samples analyzed in total displaying a relative variation between 1.08 wt% in “C1” and 3.41 wt % in “WD1”. The content of sulfur is very low, so the use of gypsum as the main ingredient of the mortars can be excluded. The low quantities of sulfur detected could be attributed to the volcanic rocks present in the island of Aegina. Similar analysis of volcanic soil from the Kyushu region, Japan has also resulted in the detection of SO_3 but in fair lower amount between 0.06 and 0.65 wt% when compared with the values found in the mortar samples from Kolona(Sumartini et al., 2019). Although the connection between SO_3 and the volcanic soil of Aegina seems a reasonable interpretation, there may also be another possible and direct link between SO_3 and the use of the large jars that cannot be ignored and it will be further discussed in the final chapter of the current paper.

This differentiation in the quality of the mortar is shown clearly in the scatter plot presented in fig. 15, where two clusters of micro-samples are clearly formed:

- A Ca-rich group, including samples P1, P3, Large jar 1, Powder 1 and Powder 2, which appear to be fine lime mortars, well mixed with small quantities of Si-rich aggregates.
- A Si-rich group, including the samples “WD1”, “WD2”, “C1” and “C2”, which appear to be coarser lime mortars, poorly mixed with a large quantity of the Si-rich aggregates.

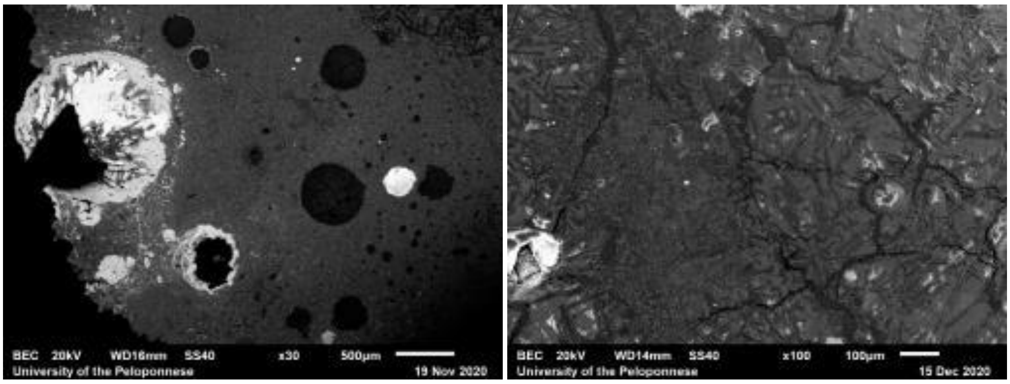


Fig. 14 The microstructure of “C1” and “C2” is shown in the SEM images above. The white rounded spots are copper-rich residues.

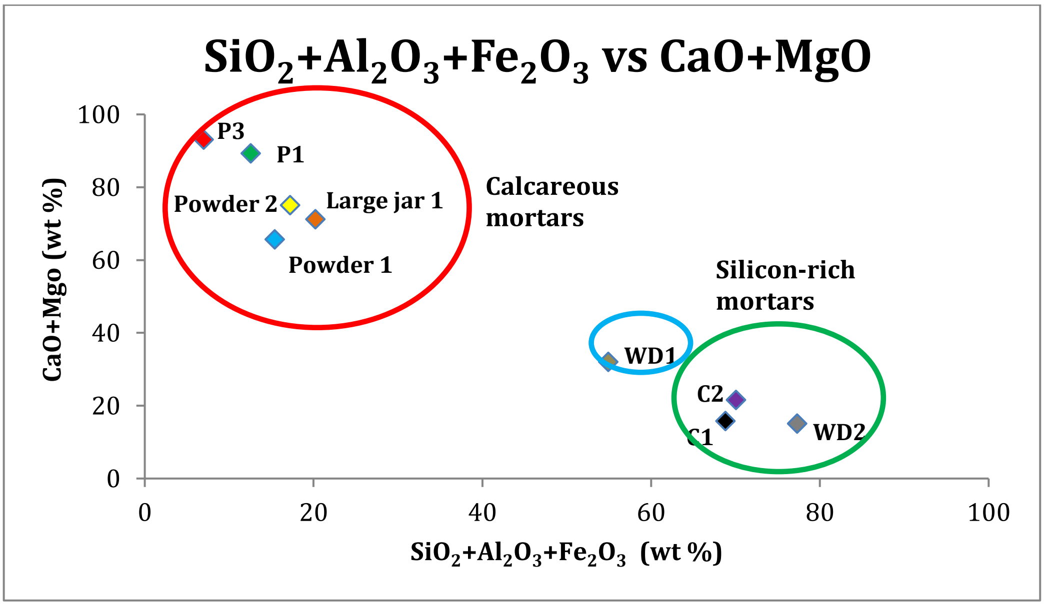


Fig. 15 The scatter plot presents two groups of mortars based on their concentrations in $SiO_2+Al_2O_3+Fe_2O_3$ vs CaO+MgO.

Tracing metal residues on mortar material is something uncommon. However, the analysis of the samples "C1" and "C2" with SEM-EDS also allowed us to study the impact of copper - that was visible even prior to the SEM-EDS analysis - on both their microstructure and composition. Residues of oxidized copper have already been presented in the SEM images of the relevant mortar sample above (fig. 14). Nevertheless, the analysis brought into light four larger areas on the samples surfaces - three in "C1" and one in "C2", where the presence of oxidized copper is much more extensive. As we can see in table 5 there is strong variation in CuO rates between 20.89 and 78.79 wt % with the oxidation of copper to be observed in two ways: 1) through the increased Cl^- content in "C1" (area 1 & 2) that reaches 14.56 and 13.49 wt % respectively and 2) through the SnO_2 values in 'C1" (area 3) and "C2" (area 1) that have significantly exceeded those of CuO. Furthermore, the absence of SnO_2 in "C1" (area 1) could be considered as another sign of copper oxidation in that area(Kantarelou et al., 2015). Fig. 16 constitutes a characteristic case of the most oxidized copper-rich area in "C1". The area has been framed at the centre of the SEM image in fig. 16 (left), where the color varies from white to light grey. The distribution of CuO (red) and Cl^- (green) is clearly depicted on the elemental mapping (fig. 16-right) of that area, where the oxidation of copper is shown in the yellow areas, where CuO and Cl^- coexist.

Table 5 The composition of the four Cu-rich areas found in the samples "Copper 1" & "Copper 2".

Cu-rich areas		SiO_2	Cl	K_2O	CaO	Fe_2O_3	CuO	SnO_2
C1 (area 1)	M.V.	6.33	14.56	1.43	5.73	-	72.52	-
	S.D.	2.25	9.7	-	0.63	-	6.94	-
C1 (area 2)	M.V.	1.09	13.49	-	3.46	-	78.79	4.11
	S.D.	-	-	-	1.44	-	23.17	-
C1(area 3)	M.V.	25.9	2.5	-	-	1.7	20.89	51.32
	S.D.	0.74	1.06	-	-	-	0.71	1.17
C2	M.V.	5.93	8.31	-	-	1.39	40.82	43.55
	S.D.	-	-	-	-	-	-	-

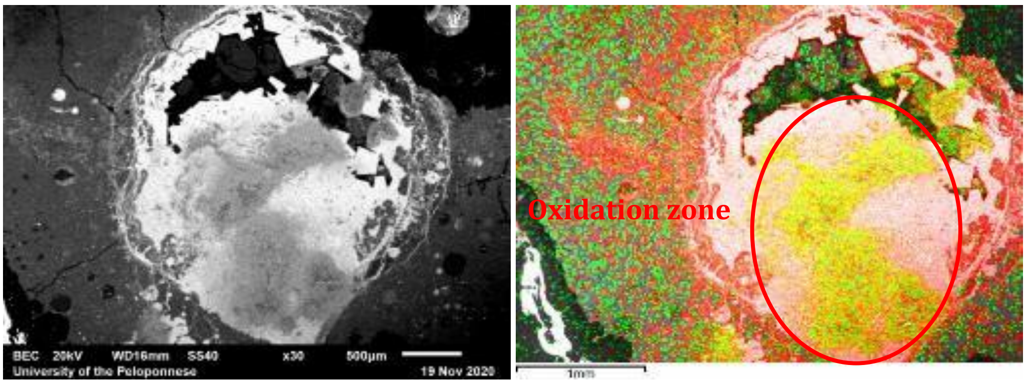


Fig. 16 The SEM images present a heavily impacted from the copper area from the sample "C1". The elemental mapping shows the distribution of CuO (red) and Cl^- (green) on the same area.

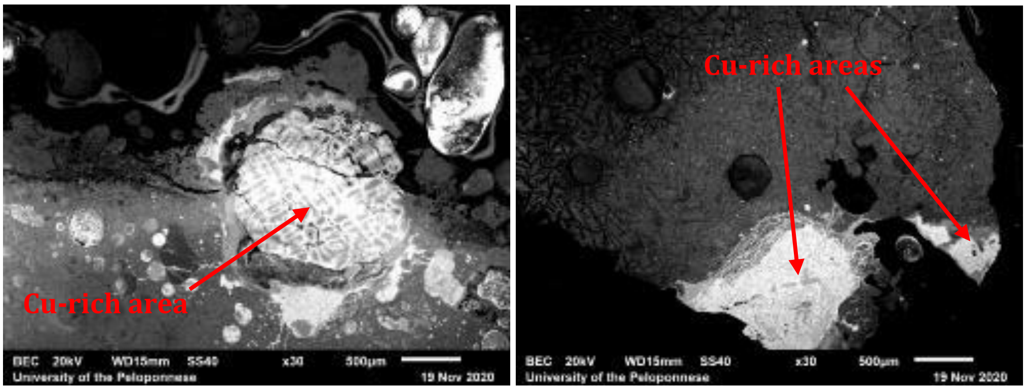


Fig. 17 The white areas shown in the SEM images above derive from copper-rich areas of the surface of "C1" (area 3) and "C2".

Apart from the oxidized copper more metal traces have been found unexpectedly in the form of brass residues in the samples "WD1", "WD2" and "Large jar 1". Several brass residues were detected at various parts of the surface of these samples as shown in fig. 18 and 19. The chemical composition of the fourteen brass residues that were analyzed in total is presented in table 6. CuO rates present a large variation ranging between 19.1 and 61.5 wt %, whereas zinc oxide (ZnO) levels are fluctuating in high levels from 31.05 to 48.35 wt %. Generally, it is known that ZnO in ancient artifacts (e.g. Roman coins, pins, swords) made of brass is found in significantly lower amounts in comparison with the brass residues found here. For instance, the analysis of 77 Roman brooches (pins) found in Slovenia with the use of Energy Dispersive X- ray Fluorescence (EDXRF) technique has led to the detection of zinc from 3.1to 21.7 % along with 76 - 90 % Cu (Istenic J., 2007). In another case study six brass coins that were dated to the 1st c. BC were analyzed through the use of Proton - Induced X-ray Spectrometry (PIXE) with zinc content fluctuating between 2.93 and 18.9 % and copper from 77.3 to 96.6 % (Hajfar H., 2015). Therefore, the increased ZnO in the mortar samples from Kolona could be considered as the result of brass oxidation as is the case with the increased SnO_2 content in "C1" (area 3) and "C2" that exceeds that of CuO (see table 5).

The overall distribution of metal traces found on the mortar samples "C1", "C2", "WD1", "WD2" and "Large jar 1" is clearly presented on the relevant pie chart (fig. 20). Brass residues is the largest group making 36 % of the measured metal traces, whereas Cu-rich, Bronze and Sn-rich groups count for 33, 20 and 15 % respectively. One of the most interesting facts upon the discovery of metal traces is that the brass residues have only been detected in "WD1", "WD2" and "Large jar 1", while the rest of the metal traces have been found in "C1" and "C2", meaning that two different metal alloys - brass and bronze - have been identified in five out of nine analyzed samples.

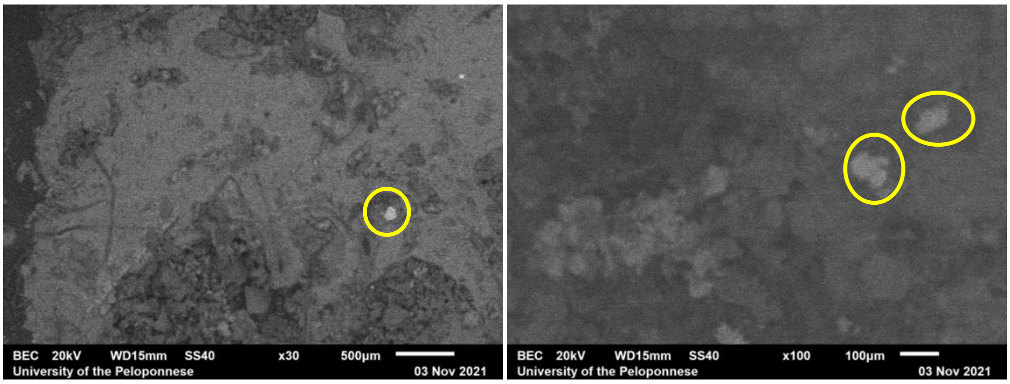


Fig. 18 Brass residues (yellow circle) have captured during the analysis if the samples “WD1” and “WD2”

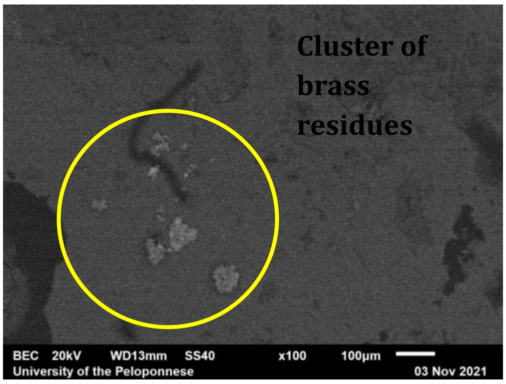


Fig. 19 A cluster of brass traces has been detected on the surface of the sample “Large jar 1”

Table 6 The table presents the chemical composition of 14 brass residues that were found on “WD1”, “WD2” and “Large jar 1”.

Sample	**Brass Inclusions**	**Na_2O**	**MgO**	**Al_2O_3**	**SiO_2**	**SO_3**	**Cl^-**	**K_2O**	**CaO**	**Fe_2O_3**	**CuO**	**ZnO**	**Total**
WD1	Brass 1	n.d.	n.d.	n.d.	31.68	n.d.	0.83	n.d.	1.52	n.d.	34.92	31.05	100
	Brass 2	6.17	0.5	1.85	39.02	4	3.77	n.d.	24.92	n.d.	19.77	n.d.	100
WD2	Brass 1	n.d	n.d	n.d	n.d	n.d	n.d	n.d	2.45	n.d	58.81	38.73	100
	Brass 2	n.d	n.d	n.d	n.d	n.d	n.d	n.d	1.4	n.d	57.32	41.28	100
	Brass 3	n.d	2.55	n.d	n.d	n.d	n.d	n.d	n.d	n.d	54.03	43.42	100
Large jar 1	Brass 1	n.d	n.d	n.d	18.39	n.d	n.d	n.d	21.44	n.d	60.17	n.d	100
	Brass 2	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	61.5	38.5	100
	Brass 3	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	51.65	48.35	100
	Brass 4	n.d	n.d	n.d	n.d	n.d	n.d	n.d	3.38	n.d	53.92	42.7	100
	Brass 5	n.d	n.d	n.d	n.d	n.d	n.d	n.d	2.88	n.d	54.92	42.2	100
	Brass 6	n.d	n.d	n.d	n.d	2.32	n.d	n.d	3.27	n.d	53.9	40.51	100
	Brass 7	n.d	n.d	n.d	n.d	n.d	n.d	n.d		n.d	57.34	42.66	100
	Brass 8	n.d	n.d	n.d	n.d	n.d	n.d	n.d	6.1	n.d	57.37	36.53	100
	Brass 9	n.d	n.d	n.d	3.55	n.d	n.d	n.d	1.01	n.d	53.42	42.02	100

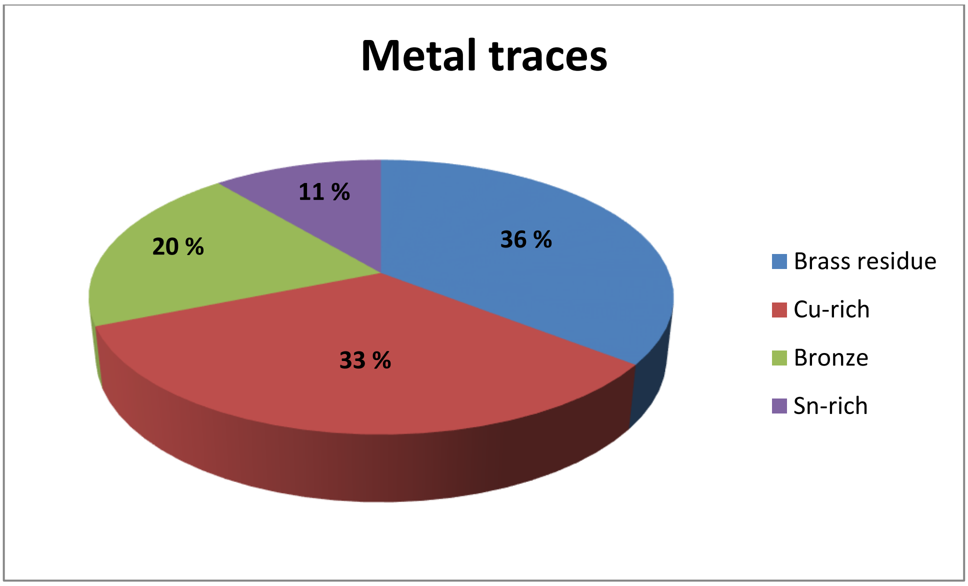


Fig. 20 The discovery of metal traces during the SEM-EDS analysis has led to the creation of four distinct groups of metals as shown in the pie chart above.

iib) Salt-rich areas

As mentioned above the proximity of the establishment, where the large jars were excavated at, to the sea shore of the island of Aegina is the main reason that explains the increased content of sodium oxide (Na_2O) and chloride (Cl^-) - the two major compounds of sea salt that were detected on several areas during the analysis of the mortar samples with SEM-EDS. The composition of six areas abundant in salt that were found on the surface of five out of nine analyzed samples is presented in table 7. Na_2O and Cl^- are the two major compounds that were traced with their rates spanning between 14.82 wt % ("WD2") and 41.01 wt % ("P1") and from 15.87 wt % ("WD2") to 39.61 wt % (Powder 1/grain 3) respectively. The highest salt content was measured in the sample "P1", whereas the elevated Na_2O and Cl^- rates in "Powder 1/grain 2" allow the description of that particular grain as salt grain.

Table 7 The composition of six salt-rich areas identified during the SEM-EDS analysis of the mortar samples is presented in the table above.

Sample		Na_2O	MgO	Al_2O_3	SiO_2	SO_3	Cl^-	K_2O	CaO	Fe_2O_3
WD1	M.V.	24.54	n.d.	n.d.	49.54	1.64	26.06	n.d.	0.57	n.d.
	S.D.	1.55	n.v.	n.v.	2.58	0.12	2.39	n.v.	0	n.v.
WD2	M.V.	14.82	3.98	n.d.	61.95	1.95	15.87	n.d.	0.95	n.d.
	S.D.	3.19	0.66	n.v.	0	0.11	1.23	n.v.	0	n.v.
Large jar 1	M.V.	18.16	n.d.	n.d.	20.44	n.d.	23.13	n.d.	36.32	n.d.
	S.D.	4.1	n.v.	n.v.	10.1	n.v.	2.74	n.v.	2.38	n.v.
P1	M.V.	41.01	n.d.	n.d.	2.22	n.d.	38.31	n.d.	19.83	n.d.
	S.D.	2.15	n.v.	n.v.	0.72	n.v.	2.32	n.v.	1.38	n.v.
Powder 1 (grain 2)		33.2	1.37	3.62	9.14	3.3	39.61	0.29	9.47	n.d.
Powder 1 (grain 3)		11.5	3.26	4.14	11.72	1.46	n.d.	13.81	52.44	1.7

To further study and understand the extent of the sea salt diffusionwithin these mortar samples, mapping was also carried out apart from the conventional bulk analysis of the mortar material. Therefore, the same areas from “WD1”(fig. 21), “WD2” (fig. 22) and “P1” (fig. 23) that were analyzed with bulk analysis (table 7), were also scanned targeting at mapping the distribution of Na_2O (red) and Cl^-(green) on the surface of these areas. The selected area from “WD1” is heavily corroded from the salt, which is mainly concentrated on the yellow-colored areas, where both Na_2O (red) and Cl^- (green) have been detected. Similarly the surface of “WD2” is also impacted by the sea salt action as shown in fig. 22, although the yellow area is not as much dense as it is in “WD1” (fig. 21) suggesting that the salt rich areas are more sparsely distributed on the sample surface. Another area from the sample “P1” was also mapped for the same reason. The marked surface shown in fig. 23 (red circle) was the only one rich in salt, but this is enough to illustrate the corrosion caused from that substance since it has covered a relatively wide area of “P1” surface. So, the elemental map depicted in fig 23 (right) shows the presence of a salt zone, where the highest amount of salt has been traced shown with yellow color.

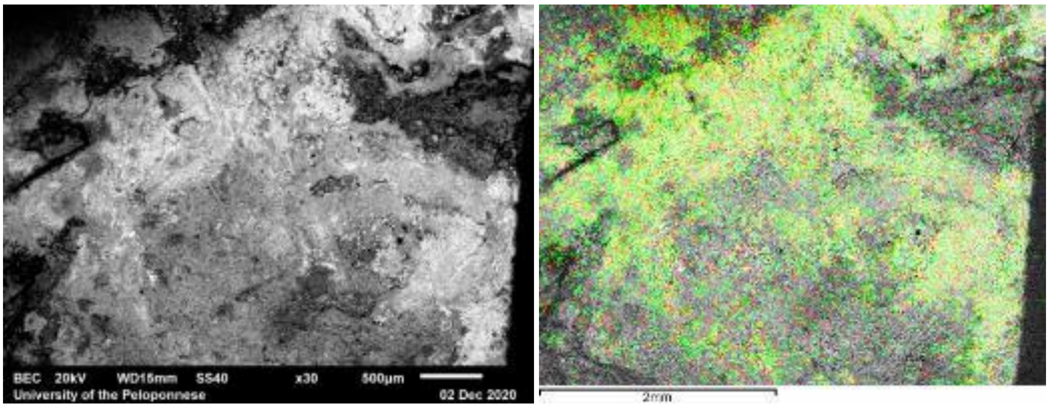


Fig. 21 A salt - rich area derived from the sample “Wall D1” is shown in the SEM images above. The distribution of salt (yellow) is presented on the mapping illustrating the distribution of Na_2O (red) vs Cl^- (green).

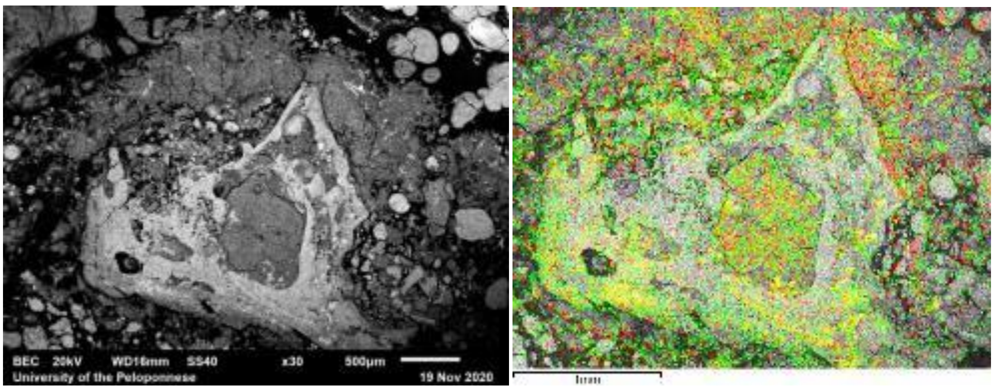


Fig. 22 During the analysis of the white plaster residues on the sample “Wall D2” (left) high amount of salt was also traced, the spread of which is shown with yellow color on the mapping (right).

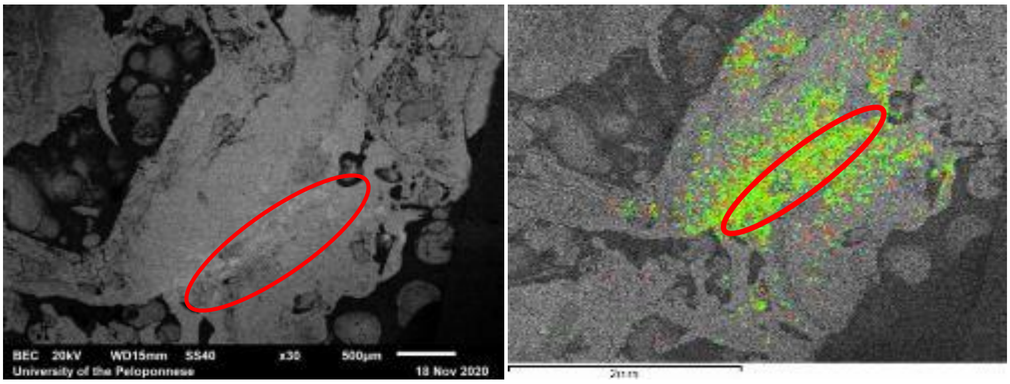


Fig. 23 Another salt-rich area has captured after the chemical analysis of the sample "Pithos 1" (left). The mapping of Na_2O (red) vs Cl^-(green) presents the salt's distribution (yellow) over the scanned area (right).

iic) Aggregates

Another key issue, on which the SEM-EDS analysis shed light, is the composition of the aggregates that were bound on the mortar material and revealed a lot of information about the nature of the raw materials used for the mortar synthesis. It is also necessary to clarify that no aggregates were found in the samples "Powder 1"and "Powder 2", because of their powdery form. As we can see in the pie chart below (fig. 24) all identified types of aggregates found in the mortar samples are typical of igneous rocks, which can be explained by the presence of the volcano in the island of Aegina that has affected the local soil that was used for the mortar creation (Mackenzie W., 1980). Si-rich aggregates have formed the largest group counting for 28 % of the analyzed aggregates followed by the Ca-rich aggregates (20 %). Si-rich inclusions have been spotted in all mortar samples except for "WD2", whereas Ca-rich aggregates are bound mainly in "Large jar 1", "WD2" and "WD1". Quartz (18 %) is the next largest group containing all aggregates, where 100 % of SiO_2 has been traced in them. Quartz is one of the most frequent types of aggregates usually found in igneous rocks and in our case most of quartz aggregates are evident in the sample "C1", which is rich in SiO_2 (53.53 wt %). Salt grains (13 %) are actually another sign about the corrosion that the mortar samples have suffered from the sea salt and they are included in this pie chart to further underline the presence of salt on the mortars surface. The vast majority of analyzed salt grains - around 90 % - is present in "WD1" and "WD2" the two most heavily impacted from the sea salt samples. The aggregates that have been precisely defined are included in the rest eight groups and they are all typical of igneous rocks (Mackenzie W., 1980). Feldspars (2 %), zircon silicate (2 %), amphibole (2 %), phylosilicates (2 %) and pyroxenes (5 %) along with quartz (18 %) belong to the wide group of silicate minerals, which are commonly found in igneous and metamorphic rocks(Mackenzie et al, 1980). Quartz occasionally occurs in sedimentary rocks too, such as sandstone. Spinels (5 %) is another important identified group of minerals, in which chromite (2 %) also forms part. On the other hand, apatite derives from phosphate minerals, in which phosphorus oxide (PO_4) is one of the major compounds. In our case apatite in the form of hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) has been found only once in the sample "Large jar 1". In general, this type of mineral is considered as crucial geological or biological residue since around 70 wt % of human bones are composed of hydroxyapatite(Junqueira et al., 2003). However, the singular detection apatite in the mortar samples is not enough to suggest a possible presence of human-linked traces. A relative variation regarding the different types of aggregates found is observed in all samples more or less, although the strongest variation can be seen in the sample "Large jar 1", wherein six different groups of aggregates (i.e. Si-rich, Ca-rich, Quartz, Zirconium silicate, hydroxyapatite, spinels group, phylosilicate) have been traced. Moreover, it is worth referring to the fact that only one aggregate (Si-rich) was found and analyzed on the surface of "P1".

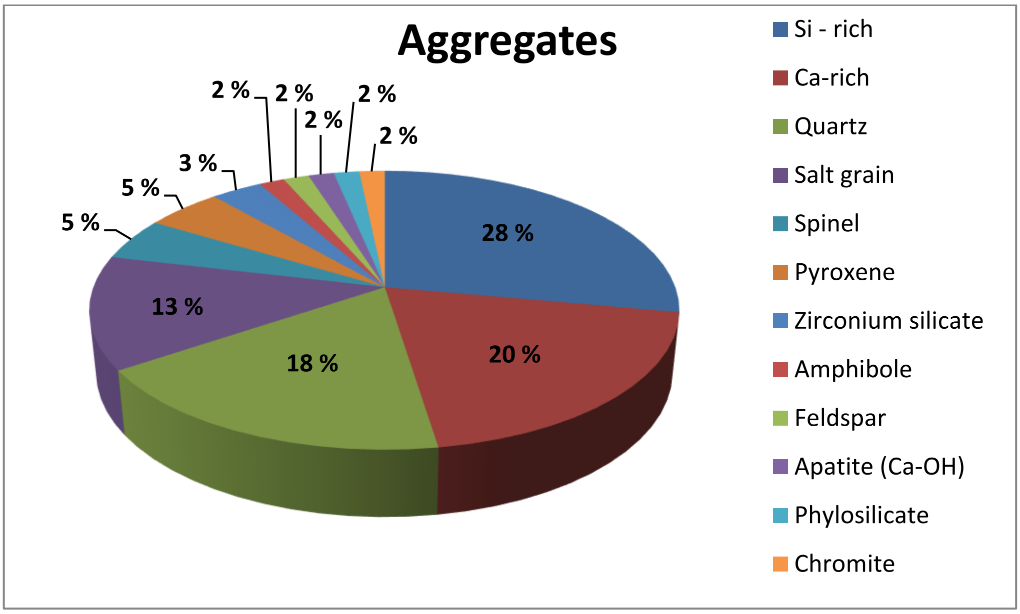


Fig. 24 The identified types of the aggregates found during the analysis of the mortar samples are shown in the pie chart above.

Lastly, the size of the analyzed aggregates is one more characteristic that is presented in the table 8. So, the minimum diameter has performed a weak variation between 10 and 30 μm, whereas the maximum diameter of aggregates fluctuates from 350 to 1700 μm.

Table 8 The table presents the minimum and maximum diameter of the inclusions analyzed in each sample.

Name of sample	Min. diameter (μm)	Max. diameter (μm)
WD1	30	350
WD2	10	1000
C1	10	1700
C3	20	900
Large jar 1	30	370
P3	10	450

Possible use of the large jars

i) The fulling process

In this chapter the main question is to evaluate the likely use of the large jars. Combining the archaeological evidence with the analysis the safest conclusion that can be reached correlates the use of that installation (i.e. large jars complex at Kolona) with the fulling process. In the Roman period (27 BC-395 AD) fulling was a very important daily procedure, the purpose of which was to clean, refine and sometimes recover woolen clothes or textiles generally (Flohr, 2009). It consisted of three main phases: soaping, rinsing and finishing, each one including several activities. The buildings, wherein the fulling process was taking place are known as fulling workshops or "fullonicae" (in latin).

During the first phase of this process (i.e. soaping) the dirty, stained clothes were immersed in a solution of hot water and alkaline chemicals used as detergents that were filling up niches carved in the floor ("fulling stalls") or large ceramic jars embedded in the workshop's floor. In particular, the detergents were alkaline - rich substances, such as animal fats or urine possessing ideal bleaching or absorbent and degreasing properties (Soriga, 2017). Once a cleaning paste had been synthesized by mixing those substances, it was first rubbed on the stained spots of the clothes in order to dissolve the dirt and then the clothes were put into the solution and remained there until they were soaked(Bradley, 2002). The laundrymen who worked in the fulling workshops, known as fullers (from the latin word "fullo"), were standing on their feet inside those niches/large jars and they were trampling the soaked clothes until all stains were removed (fig. 25). However, it is suggested by some researchers that the fullers were standing outside the niches and they were actually washing the clothes at a distance using a long thin wooden reed, in order to protect their skin from the action of the alkaline chemicals on their feet (Bercero, 2001). Once the clothes had been soaked and trampled enough, the cleaners wrung them out with their hands to remove the absorbed water. Other substances that might have been utilized for clothes washing are natron - a mixture of sodium bicarbonate, sodium sulfate and salts - potash, soapwort and the so called fuller's earth (Bradley, 2002). The latter was widely known as a cleaning agent in the past. In fact, any clay material possessing the capability to remove or decolorize oil or other liquids belongs to the fuller's earth category (Hosterman, 1992). As for urine, it was the most widely used type of detergent, because when applied on the grease on the woolen clothes, it formed a soap-like compound.

The soaping phase was followed by the second phase, during which the soaped clothes were rinsed with fresh water. Rinsing was regularly taking place in large rectangular basins, the number of which was fluctuating between one and four depending on the fulling workshop size (Flohr, 2009). The floor and the walls of the basins were coated by a waterproof plaster in order to avoid water absorption (fig. 25). The four corners of each basin were further strengthened with rims, while pipes made of terracotta or lead were constructed under the floor to provide the basins with freshwater and to remove the wastewater after the end of the rinsing process (Flohr M., 2006).

Fig. 25 Left: The above picture forms part of the lower section of the fresco found in the fullonica of "Veranius Hypsaeus" (House VI, 8, 20-21.2) in Pompeii. Fullers are shown washing the cloths by trampling on them or rubbing them with their hands (1st c. AD). Right: The interior of the basin B1 that was excavated in the fulling workshop at the House VI in Pompeii is shown in the picture above. The waterproof plaster covering the walls as well as traces of the lead pipe (red pointer) is visible.

In general, the pipe system was typically attached to the wider water system of the installation of the fulling workshop, which also formed part of the water network supply that was built under the streets of the city. During that phase the clothes were further cleaned with fresh water to rinse the detergents remained from the previous phase. If we attempt to reconstruct the whole process that was being followed, we need to take into account that the water was flowing in one direction from the first basin, where the water supply pipe ended, toward the last basin, where the sewage drain started. Thus, cleaner water would come in allowing its reuse (De Feo, 2013) so, clothes rinsing should have followed an opposite direction from that of the water, starting from the last basin, where the largest amount of the rinsed detergents were contained there (dirtiest water) and ending to the first basin, wherein the cleanest water would be contained.

Once the rinsing phase was completed, the finishing process followed. That was the most crucial phase consisting of several activities, such as drying, sulfuring, brushing, shearing and pressing, all of them aiming at the clothes refining. The wet clothes, after the end of the rinsing phase, were hung up on horizontal wooden bars probably in an open space of the fulling workshop, such as a backyard or on the roof of the building, and aired until they got dry (Flohr M., 2011). A typical scene of the clothes drying can be seen on the fresco found in the fulling workshop of "Veranius Hypsaeus" in Pompeii (fig. 26) allowing us to acquire a clear picture about drying. Three workers are depicted hanging the rinsed clothes on horizontal bars made of wood before the succeeding activities of the third phase took place. Both brushing and shearing should have been taking place almost at the same time in order to trim the clothes fibers and improve their appearance and their texture as well (fig. 26). Thistle heads were usually used as combs to raise the clothes nap (a thin layer of interlaced wool fibers) and the raised nap was trimmed until the clothes or textiles obtained a smoother surface (Bradley, 2002). This has been described as a very significant activity as it contributed to the maintenance of the nap's good quality resulting in smoother and warmer garments after the end of the fulling process (Flohr M., 2011).

Fig. 26Left: The section of the fresco depicts a scene from drying process showing workers hanging the already washed clothes on wooden horizontal bars (1st c. AD). This fresco derives from the fullonica of Veranius Hypsaeus in Pompeii (House VI, 8, 20-21.2). Right: This is the upper section of another fresco from the fullonica of Veranius Hypsaeus in Pompeii depicting workers treating the clothes during the finishing phase. On the left a worker brushes a cloth to raise the nap, while a woman with a young child inspect the processed (finished) clothes. On the right a man carries on his shoulders a curved wicker frame, over which the clothes will be placed in order to be bleached with burning sulfur. The bucket that the same worker holds probably contains the bleaching substance (1st c. AD).

A similar purpose must have been served by the sulfuring process. This was the following activity aiming at whitening the woolen clothes and making them look brighter as their white color had faded due to the fiber friction caused during the soaping phase. According to another fresco from the fulling workshop of "Veranius Hypsaeus" in Pompeii, depicting the clothes sulfuring (fig. 26), a curved wicker frame was placed above a pit, wherein sulfur (brimstone) was slowly burning. The brushed and sheared clothes were spread all over that frame in both sides in order to be bleached by brimstone fumes and to acquire the desired bright white color (Bradley, 2002). The importance of sulfuring is also suggested by the fact that there are several references in the ancient texts about sulfuring. Pliny the elder mentions in his work "Natural history" ("Naturalis historia", XXXV, 196-198) that "the cloth is first washed with earth of Sardinia, and then it is fumigated with sulfur". The "earth of Sardinia" was probably a type of fuller's earth derived from the island of Sardinia. But apart from that, it is also reported that sulfur has the potential to reveal whether the clothes were colored with a bad or good dye. In the case of a bad dye the color is degraded and spread all over the cloth by the action of sulfur (Pliny the Elder, Natural History, XXXV, 198). Once sulfuring was done, clothes may have once again been brushed and sheared and they were then pressed (fig, 34) and returned clean and folded back to their owner.

ii) Excavated fulling workshops in Pompeii and Ostia and the importance of fulling in the textile economy of the Roman Empire

Based on the aforementioned detailed description of the fulling process it is obvious that fulling was a very tiring and time-consuming procedure, the contribution of which was decisive for the clothes/textiles good quality maintenance. But, the complete and in-depth comprehension of what fulling actually was cannot solely be based on a mere description of the activities taking place in each phase. In this unit our attention will be focused on the position that fulling had in Roman society and economy as well based on information derived primarily from the architectural characteristics of the "fullonicae" excavated in Pompeii, Ostia and elsewhere so far.

Generally, the vast majority of fulling workshops has been excavated so far in Italy (Pompeii, Ostia, Herculaneum, Rome, Florence) and they are all dated in the Roman period. The most well-known examples come from Pompeii and Ostia, two well-preserved Roman cities, where a significant number of "fullonicae" of various dimensions has been found. But how can the ruins of an ancient building be recognized as fulling workshops? Moeller in his work about the fulling workshops in Pompeii relied on the presence of anything similar with large washing vats or treading stalls to identify a possible "fullonica" (Moeller W.O., 1976). However, such criteria should be regarded not only as vague but also as uncertain. In any case, they are not sufficient for any researcher to safely characterize an installation as "fullonica" (Flohr M., 2011). So, the architecture and mainly the arrangement of the interior space of those establishments are strongly correlated to the activities taking place during clothes washing and refining that allow the identification of an establishment remains as fulling workshop. Around twelve small-scale fulling workshops have been excavated at Pompeii together with some other larger at Ostia, Rome and elsewhere – allowing us to better understand their architectural characteristics (Flohr M., 2011). Several fulling workshops were established in tabernae (i.e. a type of shop in ancient Rome), while there were medium-sized fulleries that were integrated in private Roman houses (Flohr, 2009). This is the case in Pompeii, where several small or medium-sized fulling workshops have been brought to light. The "fullonica" found in Taberna V constitutes a typical case of a small-scale fullery that was actually part of a wider shop.

As we can see (fig. 27) three "fulling stalls" S1, S2 and S3 were carved on the shop floor alongside its western wall. Niches S1 and S2 are encircled by two walls, one south of S1 and another north of S2. The existence of these walls were typical in most of the fulleries found so far facilitating the job of the fullers during the soaping phase, as they were able to retain their balance by leaning on those walls, whilst they were trampling the clothes with their feet. Fulling stall S3 was later added at the north side of that complex (north of the fulling stall S2) in order to increase the capacity of clothes that could be washed (Flohr M., 2008). Between the stalls S1 and S2 there is a hole, wherein an amphora probably containing the alkaline detergents used for clothes soaping is believed to be placed (Flohr M., 2008). In the room at the northwest corner of the shop (fig. 27) a drainage installation was discovered, which is thought to be related to the fulling workshop function, although the real use of it still remains unclear. However, Flohr suggests the presence of a small water basin in that room that might have been used for the clothes rinsing (Flohr M., 2008).

Fig. 27 Left: The picture shows the three fulling stalls excavated at the west part of taberna V in Pompeii. Right: This is the northwest room of "Taberna V", where drainage installations have been excavated at.

Another case of a medium-sized fulling workshop was the one that was found in the House VI (8, 20-21.2) in Pompeii (fig. 28). This is a larger installation compared with the aforementioned small fullery. Seven fulling stalls (S1, S2, S3, S4, S5. S6 & S7) and four rectangular basins (B1, B2, B3 & B4) were forming part of that fulling workshop constructed at the west flank of that house. The first six fulling stalls were found together at the northwest corner of the house east of the basins B3 and B4 (fig. 28-green circle), whereas the seventh fulling stall (fig. 28) that was made of travertine was carved at a separate location and was attached near to the northeast corner of the basin B1 at the corridor E, few meters away from the rest of the stalls (Flohr M., 2007). According to Flohr two explanations can be given about this peculiar characteristic of that workshop (Flohr M., 2007): 1) once the clothes had been soaped, they were checked out for any stains that might have not been removed and if necessary they were soaped again in the seventh fulling stall, 2) additional treatment with cleaning agents might have been required before their rinsing in the basins. The remains of that "fullonica" provide us with a good picture about how the rinsing complexes in fulling workshops looked like. The rinsing complex of the fulling workshop in the House VI at Pompeii was mainly composed of three basins (B1, B2 & B3), while a fourth smaller one (B4) was added to its north side. Waterproof plaster was coated on the floor as well as the walls of the basins, while their corners and angles were strengthened with the rim, as described above (Flohr M., 2008). It has been estimated that B1 has a capacity of 4.100 liters of fresh water supplied through a lead pipe (Flohr M., 2008). In spite of the absence of any traces of such pipe, its imprint is still visible on the east wall of that basin revealing its existence (fig. 25). On the other hand, basins B2 and B3 had a slightly larger surface of 6,10 m^2 (B1 has a surface of 4.45 m^2), but significantly lower water capacity of about 3.500 liters (Flohr M., 2008). According to the research conducted on that complex, basins B2 and B3 (fig. 29) should have been dependent on basin B1 for their water supply. The amount of water required to completely fill up the basin B1 would be sufficient to fill the basins B2 and B3 up until the height of the steps (35.5 cm) constructed in their interior (Flohr M., 2008). This is almost half of the overall depth of each basin (60 cm) meaning that half of their capacity (1700 - 1800 lt) could have been filled. Thus, the water in basin B1 should have been refreshed at some point with a certain quantity of fresh water through the lead pipe. Basins B2 and B3 are connected to each other by a terracotta pipe opened through their separating wall, while each had its own discharge drain at their southeastern corner, through which the wastewater would have been discharged out of the fulling workshop. Generally, all discharge drains found at the southeast corner of the four basins would certainly have been covered by a metal or stone stopper, while the basins were being used. As for the fourth basin B4 (fig. 29), this seems to not directly belong to the whole rinsing complex, suggesting that it was later added to the rinsing complex. Its surface was fairly smaller (3.5 m^2) compared with the other three basins, whereas its capacity is still unknown (Flohr M., 2008). The floor of that basin clearly slopes towards the discharge drain at the southeast corner. But the fact that no traces of any pipe were found, through which the water would be flown into that basin suggests that the amount of water contained in it was very low (Flohr M., 2008). The precise function of that basin has not been defined yet.

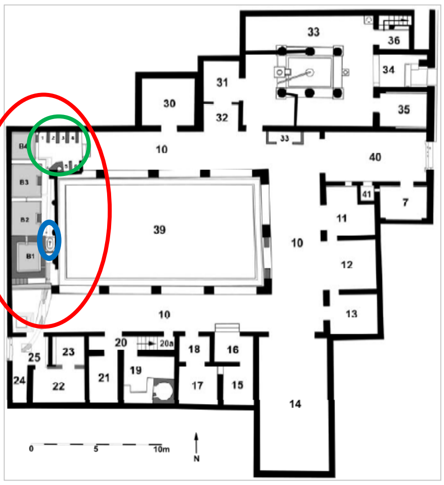


Fig. 28 The plan illustrates the layout of the House VI (8, 20-21.2) in Pompeii. The fulling workshop is outlined by the red circle with the green circle showing fulling stalls 1-6 and the blue circle fulling stall 7.Right: This is the fulling stall 7 near the basin B1 in the corridor E (right).

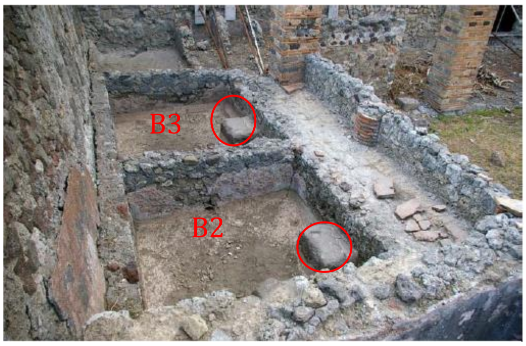


Fig. 29 Left: Rinsing basins B2 and B3 are depicted in the picture above. There is a step (red circle) at the SE corner of each basin. Right: The interior of the rinsing basin B4. The discharge drain is pointed by the red arrow.

Remains of a very large fulling workshop - the fullonica V (or fullonica della Via degli Augustali) have been excavated in Ostia, where that building seems to have been designed especially for the fulling process (Flohr M., 2009). The most characteristic feature was the dependence of the roof construction on the rinsing complex position (Flohr M., 2009). In particular the roof was sloping toward the basins so as to allow the rainwater to drop into them. As we can see in the workshop's layout (fig. 30) 36 fulling stalls were built alongside the north, west and south walls, while two of them (stalls 35 & 36) have been constructed between the basins 2 and 3 resembling the location of the seventh fulling stall in the House VI in Pompeii. In total, four basins were built at the centre of the workshop - as is the case in the previous middle - sized fulling workshop - placed one next to each other from east to west (Flohr M., 2009). Two presses, where the washed and treated clothes were folded before they were given back to their owner, were found at a separate area (fig. 30) at the southeast corner of the establishment suggesting that clothes refining (i.e. brushing, shearing, sulfuring and pressing) was taking place there during the finishing process (Flohr, 2009). In fact, the character of this fulling workshop is absolutely differentiated from that of the other two “fullonicae” since no other activities apart from fulling should have been taking place in that workshop. There were not any living quarters established in that building, where the administrator of the “fullonica” and the staff could have lived in (Flohr M., 2009).

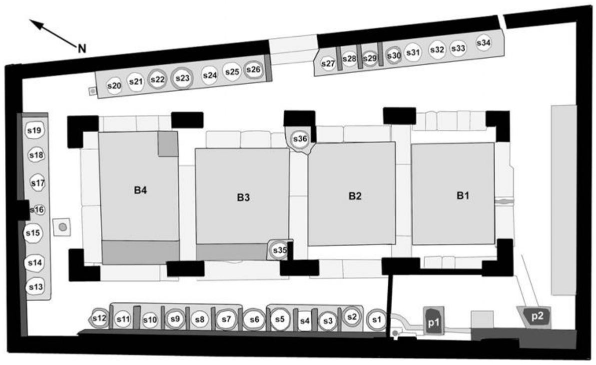


Fig. 30 The plan of the “fullonica della Via degli Augustali” in Ostia is shown above. The area were the clothes were refined is indicated by the green circle.

Depending on the archaeological research made on the fulling workshops in Pompeii, Ostia and elsewhere, two types of “fullonicae” can be distinguished according to their fulling capacity and the role that they played in the trade of textile: the small-scale fulling workshops such as that established in Taberna V and the large-scale “fullonicae” built at private houses or buildings (e.g. House VI, “Fullonica della via degli Augustali”) especially designed for fulling (Flohr M., 2011). The “fullonicae” established in private houses, such as the “fullonica” in the House VI, may have somehow formed an intermediate type, with a function closer to the small-scale fulleries. Despite the fact that they are significantly larger than the small-scale fulling workshops, their construction within the household suggests that they were actually a sort of private family business specialized in the clothes washing and refining and their operations were dependent on a private clientele just like with the small-scale “fullonicae”. As it has already been stated several small-scale fulleries have been excavated mostly at Pompeii. Of the twelve “fullonicae” found there so far, eight were built in shops (i.e. “tabernae”) with a low number of fulling stalls (2 or 3) resulting in a significantly limited fulling capacity (Flohr M., 2011). Furthermore, the direct contact of those fulling workshops with the streets of Pompeii may dictate that their function relied on a limited private local clientele that would have only derived from Pompeii and not from anywhere else (Flohr M., 2011). Three out of the twelve fulling workshops had a relatively higher fulling capacity of 5 to 10 fulling stalls and rinsing basins with two of them having been constructed in a “taberna” as well. On the other hand, the “fullonicae” found at Ostia, Rome and elsewhere are far larger than those in Pompeii (Flohr M., 2011). So it may be suggested that they were serving a broader and more professional client network derived from several cities of the Roman empire; while fulling was not really part of the textile production, textile traders could decide to have the clothes/textiles that they bought from the weavers fulled (i.e. washed and refined) in any of the fulleries in Ostia or Rome before selling them on the market (Flohr M., 2011).In general, clothes/textiles fulling should have constituted one of the pillars that synthesized the textile trade in the Roman Empire. However, according to the evidence available from the archaeological survey conducted on several Roman "fullonicae", fulling should better be taken as a service rather than a manufacturing process (e.g. weaving or spinning). In any case it seems that fulling possessed its own position in the trade of textile, as a supplementary but yet important process that may have taken place before the clothes were sold on the market (Flohr M., 2011).

iii) The location of the fulling workshops and their water supply system

The exact location selected for the fulling workshop construction in the ancient cities is another important key issue about the fulling process and how people perceived that kind of activity in general. Extensive investigation of the fulleries in Pompeii has shown remarkable and surprising facts. Although it would be expected that the fulling workshops would have been built in remote areas outside the city walls, such as the tanneries in the ancient city of Athens, the excavations at Pompeii proved that the fullonicae were built at locations inside the city complex, as shown in the map below (fig. 31). Generally, all major fulling workshops were built on the main streets of Pompeii, while the same pattern is observed in Ostia as well (Bradley, 2002). The large fullonica in Ostia is located at the rear of the large and richly-decorated "Headquarters of the Augustales" inside the city, which is considered as a prestigious and religious building (Bradley, 2002). In some cases, however, the fullonicae were situated not only in the city centre, but also next to the richest private houses or prestigious constructions (Bradley, 2002). The interior of the fulling workshops certainly stank, if we take into consideration the dirty clothes that had to be washed as well as the malodorous detergents (e.g. urine) that were used for that purpose and of course the clothes bleaching with sulfur. Nevertheless, people were not prevented from constructing fulling workshops inside the cities, despite the bad smells. Looking carefully at the remains of the fulling workshops found in Pompeii, Ostia or elsewhere and especially those established in "tabernae" the areas where the clothes were washed and treated were usually located in the back part of the shop (i.e. "taberna") or house at a considerable distance from the streets(Flohr M., 2017). So, bad smells were indeed produced during the fulling process, but it seems that not much nuisance was able to spread in the urban environment at the time of the clothes/textiles washing and refining (Flohr M., 2003). The majority of references regarding the bad odors caused by the fulling process have their routes in the jokes derived from the comedies written by Latin comedians, such as Plautus or the epigrams of Martial that overemphasize the proverbial malodorous process of fulling (Flohr M., 2003). Moreover, the locations of the major fulling workshops in Pompeii as shown in the map below (fig. 31) may have not been selected randomly. Their proximity to the public baths (B) as well as the public latrines (L) may be related to the laundrymen necessity of having access to places, where urine, the most important cleaning agent, could be collected for the clothes/textiles washing(Bradley, 2002). But this point of view is opposed by Flohr's point of view considering the "fullonicae" distribution near the public baths and latrines coincidental, because in most latrines human excrements (i.e. urine and faeces) were landed in a trough filled with flowing water that took them away to the urban sewage system rendering the collection by users impossible (Flohr M., 2003).

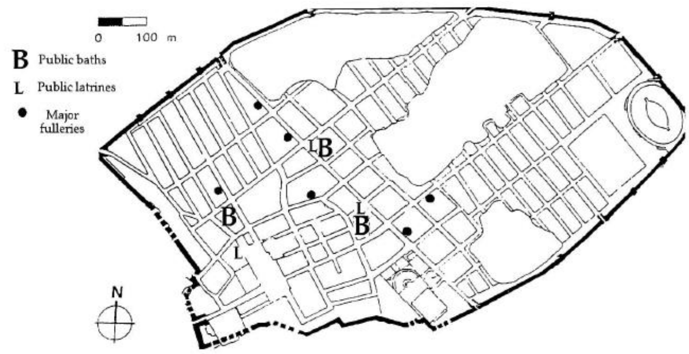


Fig. 31 The map depicts the ancient city of Pompeii, on where the exact location of the major fulling workshops (black dots), the public baths (B) and public latrines is shown.

Another important parameter impacting the fulling workshops proper function was the water supply. Most workshops and especially the medium-sized and large fulleries were directly connected to urban water supply system, whereas the small-scale fulling workshops were supplied with water from the city cisterns or fountains (Flohr M., 2006). Some other fulling workshops established in private houses were supplied with the rainwater collected by the atrium (De Feo, 2013). As it was mentioned above, water was usually transported into the rinsing complexes via lead or terracotta pipes. The majority of rinsing complexes were fed with water from one point and there was only one outlet at the opposite side of them (Flohr M., 2006) or each basin had its own drain, through which the wastewater was discharged, as is the case in the House VI in Pompeii. Therefore, this means that a rinsing complex could have either a stagnant or a flowing water system to fill up the basins. In the first case the water was stagnant and had to be refreshed, while a continuous flow of fresh water would exist in the second case. Since more than one rinsing basins have been found in the fulling workshops so far, a flowing water system seems to better match with the basins construction, allowing us to assume how the clothes should have been rinsed properly (Flohr M., 2006). Once the soaped clothes were thrown into the basins the water would automatically become dirtier, because it would bemixed with the detergents from the soaped clothes. So, the dirty water from the first basin would flow to the second one absorbing more dirt and then to the third one and so on until it reached the last basin (Flohr M., 2006). Thus, it is suggested that the clothes rinsing may have started from the last rinsing basin to end at the first one. One more challenge for the fullers would be how to get rid of the solution of water and alkaline chemicals used to soap the clothes. In the large fulling workshops there were small gutters next to the fulling stalls, where the wastewater was collected and then it was transported to the drainage of the rinsing complex. However, at Pompeii the wastewater was mainly discharged on the streets (Flohr M., 2006) as is the case in the fulling stalls in tabernae VII at Pompeii (fig. 32).

Fig. 32 Three fulling stalls were found in Tabernae VII (2, 41) at Pompeii. The wastewater after the clothes washing was removed through the gutter (red circle) made of terracotta drain in front of the stalls. Two amphora receptors carved on the corners around the fulling stalls are pointed out by the red arrows.

iv)Fullers' status in the Roman society

Finally, one more aspect regarding the profession of "fullo" back in the Roman period is the status that fullers had in the Roman society. It is frequently stated that fullers were wealthy and prosperous individuals possessing influential position in the Roman society, but this does not seem to be the case(Flohr M., 2003). Fullers were not actually as prestigious as it was once thought. A very secure indication about the fullers' real status in the Roman society is the "epitaphs", the gravestones used to commemorate the deceased fullers (Flohr M., 2003). Several gravestones have been discovered throughout the Roman Empire with the majority of those simply being referred to the name and the title "fullo" (i.e. fuller). Thus, according to such a simple report to fullers, it seems that their position in the Roman society was mainly modest, without ruling out the existence of wealthy and prosperous fullers, who might have been the administrators of large-scale fulling workshops (Flohr M., 2003). Generally, there must have been two types of fullers: those who were working possibly as slaves in the fulling stalls and the rinsing complexes in a family business and those who were running their own fulling workshop possessing higher social status (Flohr M., 2003).

v) A fulling workshop at Kolona?

Based on the detailed description of the fulling workshops architectural characteristics and the outcome of the excavations at Kolona as well as the analysis of the mortar samples the use of the large jars found at Kolona may be linked with the clothes/textiles fulling. At first, such a correlation is suggested by the arrangement of the large jars-one placed next to the other-that strongly resembles the pits found at the small fulleries in Pompeii or the fulling stalls in the larger fulleries at Ostia (fig. 33). In the three images of fig. 33 the similarities between the pits and the fulling stalls at Pompeii and Ostia respectively and large jars at Kolona can be clearly seen. Fig. 33 (middle) shows two pits carved in the floor of Taberna VI (3,6) in Pompeii that were used for clothes/textiles soaping with their interior walls having been covered by a waterproof plaster, which has not survived (Flohr M., 2008). A long row of fulling stalls found at the large fullery in Ostia is presented in fig. 33 (right). A more careful look at that picture identifies the construction of those fulling stalls by ceramic vessels that were placed into the floor separated by walls that have survived only at a very low height. At first glance, the large jars at Kolona (fig. 33-left) present a strong resemblance with the ceramic pots excavated at Ostia, but a hypothetical reconstruction of the whole installation at Kolona as it looked like in the past, would point out the large jars resemblance with the two pits shown in the middle picture in fig 33. No matter how the large jars truly looked like, their arrangement in an array in the north-south direction can be deemed as a first indication that they were being used for the clothes/textiles washing and more specifically for carrying out the soaping phase.

Fig. 33 Left: The three large jars as they were excavated at Kolona, Aegina (© Ministry of Culture, EEA / Aix-Marseille Université – CCJ – MoMArch / EFA, photo © P. Kalamara 2020). Middle: The two pits used for clothes soaping were found at Taberna VI (3, 6) in Pompeii (middle). Right: A long row of fulling stalls from the large fullery in Ostia is shown in the picture above. Ceramic vessels are embedded in the floor of the fullery in each fulling stall (red circles). The photo was taken from the east wall.

One more characteristic that could verify our suggestion about the large jars use is the presence of the white mortar rich in CaO that was coated on the upper part of the large jars interior (today survives only in the large jars 1 & 2) as well as on the area around the large jars. According to the research conducted by Flohr in several fulling workshops in Pompeii the interior walls of the fulling stalls and the rinsing basins as well were usually coated by a waterproof plaster (Flohr M., 2008); the analysis of that plaster revealed the presence of calcium carbonate content as well as of urine and ash (Flohr M., 2008). In our case high CaO content was found after the analysis of the samples “Large jar 1”, “P1”, “P3”, “Powder 1” and “Powder 2”, where the calcium-rich mortar that was covering the large jar 1 inner upper surface was found on. Calcium oxide (CaO) rates range from 61.62 to 93.02 %. In general, the use of such mortar is considered typical not only in fulling stalls but also in rinsing complexes aiming at the creation of waterproof walls that would be capable of retaining the water or any liquid substance (Flohr M., 2008). Apart from that it has been proved that such white calcareous mortar was preferred for coating the exterior walls of the buildings in Aegina. This was the case in the nearby acropolis of Kolona and more specifically with the so called “Westkomplex” that is associated with heroes or ancestors worship (Klebinder-Gauss, 2019). The exterior walls that were made of ashlars, orthostats and unfired mud bricks were coated by white mortar (Klebinder-Gauss, 2019). So, if the large jars were indeed used for the clothes washing and refining the constructors would have to create a compact interior surface that would be durable and water resistant since water possessed a significant role in the fulling process. Thus, the application of the white mortar on the large jars interior would have enhanced the interior walls strength and resistivity to water. Additionally, according to the archaeological report of the excavations that took place at the large jar establishment the floor of the installation at Kolona and more precisely its surface in front of the large jars (i.e. west of the large jars) was covered by a mosaic consisting of a strong calcareous mortar, in which small stones and ceramic shards were mixed. It was recovered in fragmentary form with one of these fragments consisting of oval-shaped small pebbles. The discovery of that mosaic constitutes further evidence that the large jars were indeed embedded into the floor with similar mosaics having been found in many fulleries in Pompeii, Ostia and elsewhere covering the same part of the floor in front of the fulling stalls or pits. In most cases the mosaics were made of mortar and fragments of ceramic pots, a type known as “opus signinum”. It was composed of a mixture of mortar and broken tiles and then it was beaten down with a rammer (Pliny, Natural History, 35, 46). This has been defined as a very characteristic feature of the fulling workshops sometimes leading to the identification of an installation as a fullery (Flohr M., 2011). Although, a clear interpretation has not been given so far, the use of these mosaics may be attributed to their function as a separating area of the fulling stalls/pits from the rest of the fullery (De Feo, 2013).

Therefore, the remains of the large jars complex excavated at Kolona seem to belong to a small-scale fulling workshop like that excavated at Taberna V in Pompeii rather than the larger complexes found in the House VI at Pompeii or in the large fulling workshop at Ostia. The small dimensions of the fullery at Kolona did not allow the whole fulling process to be carried out. However, the arrangement of the large jars one next to the other could be considered as a good sign that clothes/textiles soaping was taking place there (fig. 33). Thus, the interior of the large jars could have been filled up with the mixture of hot water and alkaline chemicals (detergents), in which the clothes/textiles were being washed by the fullers. The large jars were wide and deep enough for the laundrymen to stand on their feet inside and trample on the dirty clothes/textiles. We should always keep in mind that the excavations at the large jars establishment are in the primary phase at the time of writing, so the building has not been excavated entirely yet.

Nevertheless, soaping phase should not be regarded as the sole activity taking place at the site. Other activities regarding the clothes/textiles refining (i.e. finishing phase) could also be taking place, once the soaping phase had ended, although no clear evidence has been found yet. In particular, clothes/textiles bleaching with sulfur could have also carried out in these large jars, mostly due to the traces of sulfur trioxide (SO_3) detected during the analysis of the mortar samples. More specifically SO_3 content varies between 1.08 and 3.41 wt % with even such a low content to be enough to be considered as a safe sign of clothes/textiles bleaching with burning sulfur in the large jar complex at Kolona, Aegina. Brimstone, a sulfur-rich rock, was usually chosen as a raw material to be burnt during that process and taking into consideration the volcanic nature of the ground of this island it would not be very difficult for the fullers to find that type of rock. So, that burning substance was being burnt in the large jars interior, above which a rectangular or possibly a curved wicker frame like that depicted in the fig. 26 was placed on. The washed clothes/textiles were spread all over the surface of that frame in order to be bleached by the fumes released from the brimstone as it was burning inside the large jars and to revive the white color of the clothes. No remnants of such a frame were discovered at the excavation site. However, it should be stressed out that it would be very difficult for traces of that frame to survive, because the frame was usually made of organic material which would not easily survive for so many years. However, the fullers at Kolona, could have possibly used an alternative way to place the clothes/textiles above the burning brimstone. In that case, a horizontal wooden bar might have been attached to the interior of the roof that was known to be covering the large jars complex in order the clothes/textiles to be hung on that bar exactly above the large jars interior, from where the fumes of the burning brimstone would be coming out. This is certainly a hypothetical suggestion, but the large jars bowl-like shape and the way of their construction - embedded into the floor - would make the application of carved wicker frames above them more possible.

A lot of concern is also raised upon whether the rest of the activities (i.e. drying, brushing, shearing and pressing) involved in the finishing phase could be carried out in such a small fulling workshop. Clothes/textiles drying did not require any special installation or equipment to be carried out. As it has already been said the clothes were usually left in an open space, such as a backyard, until they dried after the rinsing phase has ended. So, it would not be difficult for the laundrymen at Kolona to find such open space to let the clothes/textiles dry. To serve that purpose a construction specifically designed to hang the clothes on it could have been used, or if the aforementioned wooden bar did exist, it could have had another use for hanging the clothes/textiles on it as well. Brushing and shearing, two important activities that normally succeeded drying and preceded sulfuring, could have possibly included to the set of activities taking place at Kolona. The problem is that no brushing and shearing tools were found during the excavations causing problems upon whether these two activities did happened at the time of the large jars use. Lastly, clothes/textiles pressing could not be included to the activities carried out in that fulling workshop since no remains of a construction used for that purpose as shown in fig. 34 have been found.

Fig. 34 This photo was taken from the fulling workshop at Taberna VII (2, 41) in Pompeii and depicts the remains of a construction possibly used for the clothes/textiles pressing.

The final significant question that still remains unanswered is whether and where the clothes/textiles were rinsed at Kolona. No remains of rinsing basins have yet been discovered at the excavated site, therefore two hypotheses can be suggested: the first possible hypothesis is that clothes/textiles rinsing was not taking place at Kolona, at least in the form that was normally being carried out. However, that phase was very important for the clothes treating prior to their refining (finishing phase). So, it seems improbable that the clothes/textiles at Kolona were not rinsed at all. The second hypothesis is that the rinsing phase was taking place in another location, probably close to the large jars complex, but they have not been discovered yet. Certainly further archaeological investigation at the surrounding area of the large jars will bring into light more findings that will further enhance our knowledge about the use and the activities occurring in that fulling workshop.

Conclusions

Seven mortar samples were collected from the large jars installation, of which six were taken from the large jar 1 interior (i.e. samples C, WD, Large jar 1, P1, Powder 1 & Powder 2) and one from the area between the eastern wall and the large jar 3 (i.e. sample P3).

According to the samples examination with OM and LED microscopes their main color identified is "very pale brown" with several other colors or hues, such as "reddish brown", "light yellowish brown" and "greenish black" identified in specific areas as well. Some white - colored areas that were occasionally found on the surface of the samples (P3, WD) correspond to the purest parts of the mortar material. Numerous small aggregates of varying color (e.g. black, grey, red, blue etc) were also visible on the samples surface.

Nine micro-samples (C1, C2, WD1, WD2, Large jar 1, P1, P3, Powder 1, Powder 2) were extracted in order to be analyzed with SEM-EDS. The analysis with SEM-EDS showed that the mortar consists of a calcareous mortar that was made of limestone with the CaO content varying significantly between different samples (from 11.3 wt % in C1 to 93.02 wt % in P3). The low amount of magnesium oxide (MgO) and sulphur trioxide (SO_3) do not support the use of dolomitic limestone or gypsum respectively as the primary material for the creation of the mortar.

In all micro-samples the analysis revealed that the limestone was mixed with silicon-rich aggregates in varying concentrations, which enhanced the durability of the mortar. The most common types of aggregates are Si-rich minerals, such as quartz, feldspar, amphibole etc. That the sample "Powder 2" was collected from the base of the large jar 1 in conjunction with the high CaO amount (71.28 wt%) traced in it suggests that the white mortar was covering the entire inner surface of the large jar 1 and not just the upper part of the large jar interior as had been originally supposed from the archaeological excavation in the establishment.

Based on the concentrations of CaO and SiO_2 two groups of mortars can be formed: 1) one with mortar samples rich in CaO (Large jar 1, P1, P3, Powder 1 & Powder 2) and 2) another group containing the rest of the samples (C1, C2, WD1, WD2). The samples of the second group can be described as calcareous mortars of bad quality, where the amount of the Si-rich aggregates significantly exceed the CaO content.

During the analysis of the mortar samples metal traces were detected in five out of nine analyzed samples (C1, C2, WD1, WD2, Large jar 1). The analysis of these residues with SEM-EDS showed the presence of two distinct alloy residues: that of bronze (CuO+SnO) identified in C1 and C2 and that of brass (CuO+ZnO) found in WD1, WD2 and Large jar 1. Both alloys were found in oxidized form. In the case of the bronze its oxidation is visible through the increased Cl^- (8.31-

14.51 wt %) rates or the increased SnO content (above 15 wt %). On the other hand, the oxidation of brass traces in WD1, WD2 and Large jar 1 is shown through the high rates of zinc oxide (ZnO), which fluctuates from 31.05 to 48.35 wt %. The presence of the oxidized bronze and brass in the mortar samples could be explained by the possible presence of an oxidized bronze or brass metal object, which was buried for a long time period with the large jars until the excavations started, thus leaving its traces on the large jars mortar.

Lastly, the use of SEM-EDS for the analysis of the samples helped in the identification of several areas of the samples surface (WD1, WD2, Large jar 1, Powder 1 & Powder 2) rich in sea salt (Na_2O+Cl) that highlighted the extent of the sea salt diffusion on these mortar samples.

Overall, it appears that the use of the large jars is related to fulling, because of their arrangement - one built next to the other - and the use of white mortar for coating the entire inner surface of the large jars. Several investigations of fulling workshops in Pompeii have shown that calcium-rich mortar was covering the interior of the fulling stalls, wherein the clothes were washed. Thus, it is suggested that the establishment, where the large jars were found, was used as a fulling workshop for washing and refining the clothes, although a more extended archaeological survey is necessary in order to bring into light more finds that will verify this suggestion.

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