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Study and Analyses of Pigments in Minoan Larnakes from the Peripheral Unit of Rethymnon (Crete) Applying Non-Destructive Techniques: Preliminary Results

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Abstract The aim of the paper is to present the first results of the study and the analysis of the pigments, which were used for the decoration of the larnakes from the Peripheral Unit of Rethymnon (Crete), dating back to the Late Bronze Age, specifically in the LM III period (1390-1070 BC). For the analysis and the identification of the pigments on the coloured surfaces of the larnakes were used the non-destructive spectroscopic methods XRF and RAMAN. Due to the large number of the larnaкes and their extraordinarily high archaeological value, which makes it impossible to transport them, the use of portable Raman and XRF spectrometers for the analyzes carried out in the archaeological warehouse of the Ephorate of Antiquities of Rethymnon, as well as in the old Archaeological Museum and in the contemporary exhibition of the new Archaeological Museum of Rethymnon. The analysis revealed the use of a variety of pigments, both organic and inorganic, which appear in the majority of the larnax, as basic paints or as random admixtures or as oxidation of mineral paints. The preliminary results of this paper add another lost link to the ancient technology of Minoan civilization and surprise us with the use of unknown –until now- raw materials, both domestic and imported, such as the realgar, Egyptian blue etc.

Keywords Pigments, Larnakes, Minoan Civilization, Crete, Raman Spectroscopy, XRF Spectroscopy

1. Introduction

At the beginning of the LM III A1 period (1390-1370/1360 B.C.) a major increase of domestic facilities [6,11] and population growth is attested in Crete [7], a fact which comprises an all-pervading phenomenon and appears to be the outcome of an overall prosperity that characterises the island after the definite end of palatial civilization and the broader changes made in the administrative system [15]. Equivalent changes also occur as far as the increase in the number of burial sites is concerned reflecting possible changes in burial customs and practices [17] of the LM III period (1390-1070 B.C.). Cemeteries are found in a number of sites in Crete, with tomb chambers being the predominant type in the vast majority of the island's locations. Excavation data suggest grave offering of new types of vases as well as various other objects, while along with the use of wooden stretchers or biers for burial rituals, the use of clay larnakes, which was common up until the MMIII-LMI period (1700-1600 B.C), reappears [11].

The majority of the studies on clay larnakes of the LM III period are limited until nowadays to theoretical approaches, based on their typological examination, and to theoretical analyses of decorative motifs on their surfaces [9,10]. Archaeometrical research on the fabric of the clay out of which the larnakes have been made and also on the pigments which adorn them are still at a primary stage, therefore depriving information which could offer valuable insight into the technicians and the workshops where they were produced and on their transportation across Crete during that period.

In the Peripheral Unit of Rethymnon, where all the larnakes in the present study derived, individual graves and several groups of graves of the LM III period have been excavated [8,13,14], and additionally two very important organised cemeteries have been revealed (**figure 1**), where chamber tombs are mainly found: the first one in “Mezaria” site of Maroulas [13,14] at a distance of 8 km. southeast of Rethymnon (**figure 3**), and the latter in “Prinokefala” site of Armenoi [12,14,20-22] at a distance of 10 km. south of Rethymnon (**figure 2 and 4**) which consists the greatest known cemetery of the LM III A-B periods on Crete until now (LM IIIA1, 1400-1375 BC - LM IIIA2, 1375-1300 BC, LM IIIB, 1300-1200 BC).

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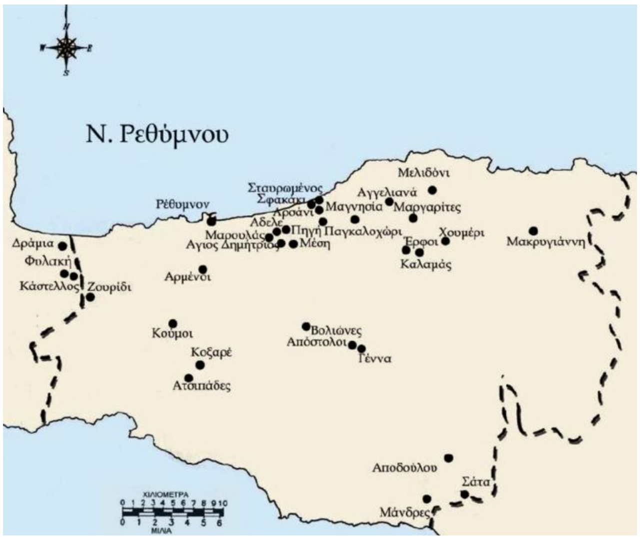

Figure 1. Map of the Peripheral Unit of Rethymnon with signed the burial sites of the LM III period [14]

Figure 2. Necropolis of Armenoi. View of the East [21]

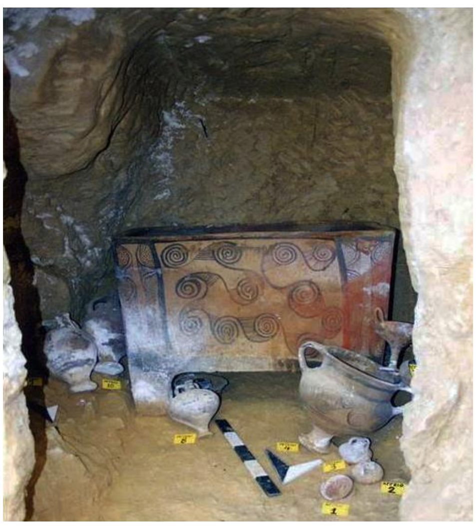

Figure 3. Necropolis of Maroulas, Tomb 7 [14]

A significant number of larnakes (more than 60) comes not only from individual graves and groups of graves but also from the two organised cemeteries (Maroulas and Armenoi) of the Peripheral Unit of Rethymnon. On their whole, the larnakes are chest-shaped, made out of coarse fabric of various shades (brown, red, reddish-brown, etc.) and are characterised by the existence of four to six legs. They also have handles on the chest as well as on the cover, while many examples of larnakes have two holes on their rim and lid through which a rope was put for making the cover more stable. At times, holes are also seen at the bottom of the chest which made the decaying of the dead bodies easier.

Figure 4. Painted LM III A2 Larnax from the Armenoi necropolis [20]

The majority of the larnakes has a white, brown, pink or yellowy coating, as well as painted decorative motifs of red/reddish-brown, black or white colour. In two examples of larnakes from the cemetery of Armenoi colourful decorative motifs of red, black, white and light gray-blue colour are still preserved. The difference in the colour of the clay and the coating, as well as in the coloured depiction of the decorative motifs is remarkable and is most likely due to the uneven firing of the larnakes [10,11].

Regarding to the painted decoration of the larnakes from the Peripheral Unit of Rethymnon, it should also be mentioned that the motifs of the LM III larnakes coincides to a great extent with the painted decorative motifs met in the vases of this period. More specifically, decorative motifs are preserved in the majority of the larnakes of Rethymnon, which could be classified into three groups: (a) abstract or geometrical motifs (spiral, chequerboard, wheel patterns, wavy lines, etc.), (b) depiction of deities or religious symbols, ritual scenes and events (double axe, horns of consecration, animal hunting, goddess with raised hands, etc.) and (c) flora and fauna dedicated to a deity (deer, birds, cows, octopuses, nautiluses, starfish, papyrus leaves, palm trees, quatrefoils, etc.) [23].

2. Experimental Work

In this study are presented the first results of archaeometrical examination on the pigments of 40 larnakes with painting decorations from the Peripheral Unit of Rethymnon, the majority of which dates back to the LM IIIA2- LMIIIB period (1370/1360-1190 BC).

The main goal of the research is to identify the types of pigments used for the adorning of the larnakes and also to discern any possible differentiations in the use of colours, which are most likely caused by the different workshop productions of the larnakes or by the different ideological perceptions of societies of the era concerning the use of colour on objects meant for burial use [4].

Regarding the choice of methods applied in the research, the following criteria were established from the outset due to the great archaeological value of the objects and their very fragmentary preservation: the methods used were to be non-destructive, the analysis was to be made in the area where the objects are stored, no sampling should be required given the poor preservation of the pigments, no time-consuming preparation should be needed, the test time would be short and, most importantly, no color or elemental corruption should occur in the analyzed pigments, in order to allow for further future testing with other methods [5].

Totally, they have been carried out 175 measurements with the use of spectroscopy with X-ray fluorescence (XRF) on the bodies and covers of the larnakes, in order to identify whether similar pigments for the decoration of the chests and their lids had been used. Meanwhile, 108 measurements were carried on the chests of the larnakes with the use of Raman spectroscopy, due to the limited time available for the carrying out of the research while only 3 lids were measured with the use of Raman spectroscopy. All measurements were held at the warehouse of the Archaeological Museum of Rethymnon with the use of portable instruments, thus avoiding the transportation of the larnakes to an archaeometric laboratory, which was literally impossible due to their impaired preservation condition, but mainly because of the huge dimensions and weight of the objects. For the examination of the larnakes with the use of XRF and Raman spectroscopy techniques, 23 hours of labour were required in total in order to complete the archaeometrical research on all larnakes and their lids [4].

Figure 5. Archaeometric examination with Raman spectroscopy

For the determination of the dye compounds, the Portable RockHound 785 spectrometer of the DeltaNu house with attached NuScope optical microscope was used. The lazer selected had a wavelength of 785 nm with a 35-micron radius, a resolution of 5 cm^{-1} and a range of 200-2000 cm^{-1}. The measurements with the use of Raman spectroscopy were carried out while holding the instrument ourselves as it was not possible to put the instrument on a basis because of the dimensions and the weight of the larnakes which did not allow for any transportation. Correspondingly, for the elemental analysis of pigments, the Skyray portable XRF EDX pocket III spectrometer was used with S (16) to U (92) data detection ranges. Also, in the case of the measurements with the use of XRF spectroscopy, they were all carried out while holding the instrument (**figure 5**).

The duration of each measurement using the RockHound 785 spectrometer was in the range of 2-5 minutes, and no Raman spectrum was then processed. The analysis of the Raman spectra was carried out using the OriginPro and Spectragryph software, while the identification of pigments with the UCL Raman Spectroscopic Library, CHSOS and RRUFF databases.

Measurements using XRF spectroscopy had a maximum of 20 seconds, and due to a technical problem, that occurred during the measurements and which we were unable to solve, it was not possible to transfer the data to a computer. As a result, the instrument display data on the PDA unit screen was recorded using a digital camera, but without the possibility of subsequent processing of the XRF spectra.

Thus, a future measurement with spectroscopy XRF is required so as to verify the results acquired from Skyray portable XRF EDX pocket III spectrometer, and also to identify data which the instrument might not have identified due to its detection abilities (**figure 6**).

Figure 6. Archaeometric examination with pXRF

3. Measuring Results

The preliminary results of the archaeometrical examination of the larnakes from the Peripheral Unit of Rethymnon with the use of Raman and XRF spectroscopy indicated the existence of various organic and inorganic pigments which were used for the decoration of the larnakes (**figure 8**).

As mentioned earlier in the introduction of the present study, the majority of the larnakes is decorated with red/reddish-brown and black or white colour, while in two examples of larnakes from the cemetery of Armenoi polychromy is observed with grey-blue colour added to the colour combination (**figure 7**).

From all the pigments identified, we consider that the white colour of the gypsum ($CaSO_4.2H_2O$) and of calcite ($CaCO_3$) was used as the basic colour of the majority of the larnakes, while possibly the colour of the white lead [$2PbCO_3.Pb(OH)_2$] was also used to a smaller extent, as ascertained by the presence of Pb by 92% in the XRF measurements. The above mentioned colours, whose distinctive peaks were identified in most of the spectra yielded by the Raman spectroscopy and which correspond with the bibliographical data, seem to have been used not only as an undercoat for the later colour decoration of the larnakes but also for creating suitable gradations of colour shades. Additionally, the white colour of the gypsum ($CaSO_4.2H_2O$), which its distinctive peaks, and has minor variations within the spectra at 418, 485, 613, 680, 1011 and 1131 cm^{-1}, might have been used as a connecting means for the solidification of the colours on the larnakes' surfaces due to its qualities (**figure 9**).

Figure 7. Polychrome Larnax (1709) from Armenoi necropolis [20]

1709	XRF Results		
	Colour/ Visual Observation	Chemical Elements detected (%)	Chemical Elements detected (ppm)
	Black (inner part of the larnax body)	Ca, Ti, Fe, Mn	Cr, Ni,Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Pb
	Red (from the body of larnax)	Ca, Ti, Fe, Mn	V, Cr, Ni,Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Sn, Pb
	White (from the body of larnax)	Ca, Ti, Fe, Mn	V, Cr, Ni,Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Sn, Pb
	Blue/Grey (from the body of larnax)	Ca, Ti, Fe, Mn	V, Cr, Ni,Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Pb
	Blue/ Grey (from the narrow side of the body)	Ca, Ti, Fe, Mn	V, Cr, Ni,Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Sn, Pb
	Red (from the lid)	Ca, Ti, Fe, Mn	V, Ni,Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Pb
	Black (from the lid)	Ca, Ti, Fe, Mn	V, Cr, Ni,Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Pb
	Blue/Grey (from the lid)	Ca, Ti, Fe, Mn	V, Cr, Ni,Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Pb

Figure 8. XRF Spectroscopy Results from the polychrome Larnax (1709)

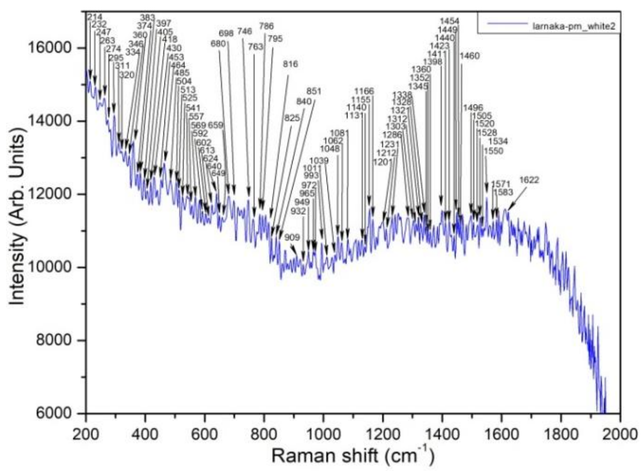

Figure 9. Raman spectra of white colour from Armenoi Larnax (1709)

The red pigments measured in the whole of the larnakes with the use of Raman spectroscopy are the red ochre (Fe_2O_3 + clay + silica), the realgar (As_4S_4) and the purpurin ($C_{14}H_{18}O_5$). All of them were identified as basic or secondary pigments for the creation of bright and solid shades of red seen on the surfaces of the larnakes, ranging from light red-orange to dark red-brownred colour. The data given by XRF spectroscopy confirmed the data of the Raman measurements, given the fact that Fe is found in all measurements, while the presence of As, a basic element of the realgar (As_4S_4), is also affirmed by 96% of the total measurements. Furthermore, it should be mentioned that purpurin ($C_{14}H_{18}O_5$) was detected in the majority of the Raman measurements with minor variations at 949, 1016, 1041, 1088, 1141, 1159, 1233, 1319, 1330, and 1448 cm^{-1}, although its validation with the use of XRF spectroscopy was not made possible given the organic nature of this pigment. The identification of purpurin ($C_{14}H_{18}O_5$) demands future measurements to be repeated with the use of both Raman spectroscopy and other non-destructive spectroscopic techniques which can detect organic pigments. Nonetheless, it should be stated that this pigment has recently been identified in analyses of wall painting sherds from Knossos [19], as well as in objects found in the Peak Sanctuary of Vrysinas which is located at a small distance southeast from the city of Rethymnon [3].

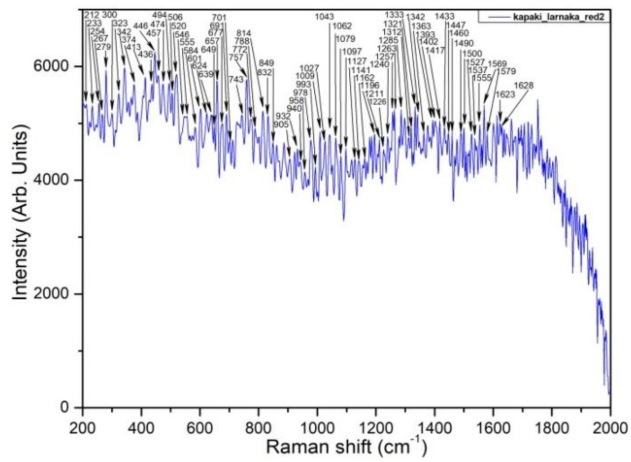

Figure 10. Raman spectra of red colour from Armenoi Larnax (1709)

The black pigment identified in almost most of the measurements with the use of Raman Spectroscopy either as a basic colour or as an admixture for the gradation of colour shades is the black colour of carbon, which is found with minor variations in almost all measurements with distinctive peaks at 957, 1328 and 1581 cm^{-1} (**figure 11**).

The blue pigments found in the majority of measurements are Azurite ($2CuCO_3.Cu(OH)_2$) and Egyptian blue ($CaCuSi_4O_{10}$). Azurite is also met with minor variations in most colours analysed at 253, 401, 547, 764, 840, 947, 1097, 1430, 1440, 1454, 1581 and 1622 cm^{-1}, mainly as either a random admixture or as a means of creating suitable colour shades. The Egyptian blue ($CaCuSi_4O_{10}$) respectively is also detected in a great number of different colour measurements, as well as in the measurement of grey-blue colour with minor variations as far as distinctive peaks are concerned at 205, 231, 362, 379, 427, 481, 568, 587, 603, 760, 767, 782, 985, 993, 1015, 1043 and 1085 cm^{-1}. The presence of Azurite ($2CuCO_3.Cu(OH)_2$) and of the Egyptian blue ($CaCuSi_4O_{10}$) in the measurements is confirmed by a great percentage of measurements and more specifically by the presence of Ca as well as Cu at a total rate of 77%. Regarding the measurements of grey-blue colour, the main pigment used has not been identified for the time being, apart from Azurite ($2CuCO_3.Cu(OH)_2$) and the Egyptian blue ($CaCuSi_4O_{10}$) which have been detected among other pigments in the measurements of the grey-blue colour (**figure 12**). Therefore, a re-examination of the grey-blue colours is required with the use of Raman and XRF spectroscopy as well as other spectroscopic techniques so as to be able to draw safe conclusions.

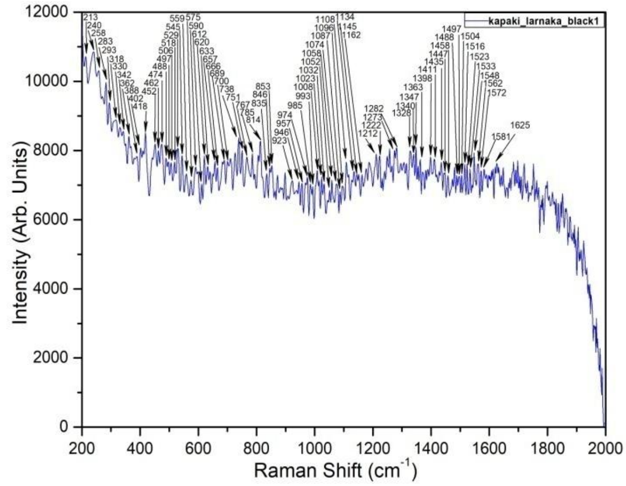

Figure 11. Raman spectra of black colour from Armenoi Larnax (1709)

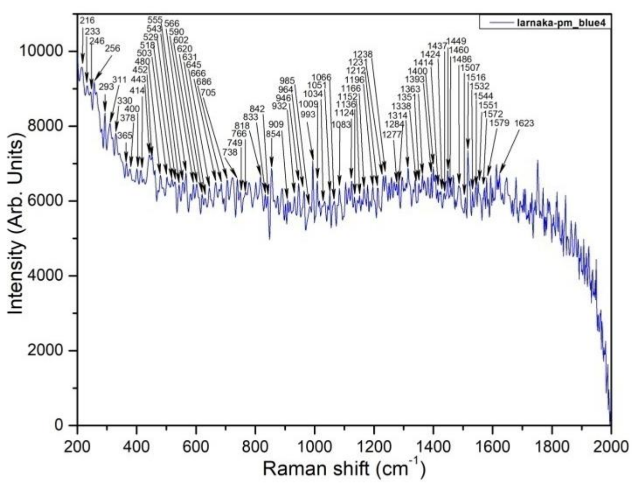

Figure 12. Raman spectra of blue colour from Armenoi Larnax (1709)

The yellow pigments identified with the use of Raman spectroscopy are the yellow ochre [($Fe_2O_3.H_2O$) + clay + silica] and saffron ($C_{20}H_{24}O_4$). The yellow ochre [($Fe_2O_3.H_2O$) + clay + silica] is found in measurements of various colours with minor variations at 237, 293, 388, 416 and 1009 cm^{-1}, while its presence is confirmed by most measurements as Fe, a basic element of ferrous pigments, is detected in all XRF spectroscopy measurements. In addition, the organic pigment of the saffron ($C_{20}H_{24}O_4$) was identified in a great number of Raman spectroscopy measurements which occurs with minor variations in all measurements at 1166, 1213, 1284 and 1532 cm^{-1}. The presence of saffron ($C_{20}H_{24}O_4$) in the pigments was not possible to be confirmed with the use of XRF spectroscopy owing to the organic nature of the pigment. Thus, further research is required, using other non-destructive spectroscopic techniques which will or will not finally verify the presence of this pigment.

At least, the green pigments which were identified as admixtures in the measurements of the red, black and blue-grey colours and also as random admixtures in the measurements of white colour are Malachite ($CuCO_3.Cu(OH)_2$) and Green Earth [$K[(Al^{III},Fe^{III})(Fe^{II},Mg^{II})],(AlSi_3,Si_4)O_{10}(OH)_2$]. Malachite ($CuCO_3.Cu(OH)_2$) is present in a great number of Raman measurements with minor variations at 216, 263, 342, 436, 505, 543, 561, 756, 1054, 1081 and 1488 cm^{-1}, while its presence is also reaffirmed by Cu identification in XRF measurements. However, it is very likely that the presence of Malachite ($CuCO_3.Cu(OH)_2$) is either associated with its deliberate mixture with other pigments for the creation of a graded red colour or it is the result of Azurite ($2CuCO_3.Cu(OH)_2$) corruption/decomposition, a pigment which is found, as mentioned earlier in the text, at a high rate of the total amount of measurements. The Green Earth [$K[(Al^{III},Fe^{III})(Fe^{II},Mg^{II})],(AlSi_3,Si_4)O_{10}(OH)_2$] is also detected as a secondary pigment in measurements of other colours, while it is met at 399, 633, 684, 816, 1002 and 1080 cm^{-1} with minor variations. Its presence was impossible to be confirmed with XRF spectroscopy, as the instrument employed could not recognise the basic elements of this pigment's composition. Nevertheless, the use of Green Earth [$K[(Al^{III},Fe^{III})(Fe^{II},Mg^{II})],(AlSi_3,Si_4)O_{10}(OH)_2$] has been known from other sites on Crete, such as Faistos (the palace area), where it has been found most probably as an admixture in wall painting for red colour gradation [16]. Thus, further research has to be carried out so as to verify whether Green Earth had been used in the colours which adorn the larnakes from the Peripheral Unit of Rethymnon for red colour gradation.

4. Conclusions

Summarizing, according to the findings of the measurements with the use of Raman and XRF techniques, is found a variety of pigments that is identified in the total number of the colours analysed. Those pigments had either been used as basic colours for the rendering of white, red, black and blue-grey or as random admixtures or, finally, as pigments of minor importance for the creation of graded shades and solid colours of appropriate opacity.

In all measurements carried out with the use of Raman spectroscopy, a likewise presence of various inorganic and organic pigments in all the measurements of red, black, white and blue-grey colours was identified. Their presence was also confirmed to a great extent by the results of XRF spectroscopy radiation. The presence of different pigments in most colours is justified by the consecutive layers of different colours, such as the one depicted in photos taken by the microscope of the Raman instrument during the measurement of the larnakes' colours (**figure 13**).

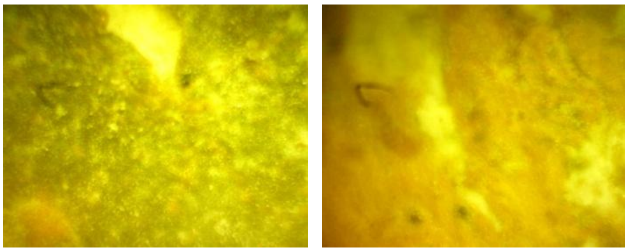

Figure 13. Blue and red colour from Armenoi Larnax (1709) with traces of other pigments

Furthermore, the presence of valuable raw materials in the colours of the larnakes should also be mentioned, such as Realgar, Azurite and Egyptian blue which are all imported. The use of Egyptian blue and Azurite on objects and wall paintings of this period on Crete is widely known [1,2,18], while the use of realgar on wall painting fragments from Knossos [19] and in pigments which decorate vases and figurines of an earlier period from the Peak Sanctuary of Vrysinas [3], Rethymnon, has been testified.

Also is remarkable the identification in the majority of measurents of the two organic pigments of Purpurin and Saffron which derived from plants growth in Crete. These pigments have been recently indicated on various objects from Rethymnon [3], while purpurin in particular has also been indicated on fragments of wall paintings from Knossos [19]. We are now aware of the fact that apart from inorganic pigments, indigenous plants which produce cheap and easily accessible pigments were also used on the island of Crete. Yet, it is essential that further research and new measurements are carried out with a view to verify the presence of these organic pigments with the use of different techniques.

Concluding, must be emphasized that the preliminary results of the non-destructive Raman and XRF spectroscopy techniques have been remarkably encouraging and in the vast majority of the measurements these spectroscopic techniques proved to be appropriate both for the determination of the pigments chemical compound and for the analysis of their elements. The scant preparation of the objects prior to the measurement, their quick analysis as well as the information drawn are but a few of the advantages of the abovementioned spectroscopic techniques, otherwise impossible with the use of techniques that require sampling. Finally, it should be mentioned that the research is still at a primary stage and that we are in hope that its continuation will offer further information on the pigments which had been used for the decoration of the larnakes and also on the identification of the workshops where they were produced.

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