Structural / Compositional Identification of Materials from Sacidava Fortress

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Abstract: *The research carried out on the Sacidava Fortress revealed a complex archaeological monument with predominantly Roman fortifications dating from the II-VII centuries AD. The investigative efforts based on advanced analytical techniques and imagistic techniques, are useful to understand the composition and levels of degradation of the Roman mortars present at this archaeological site. Analysis of the Roman mortars from this fortress revealed the presence of carbonate phases, bound water, and hydraulic compounds such as silicate and aluminate hydrates. Besides this, the microbiological analysis completed the obtained analytical data. This detailed study sheds light on the historical significance and construction methods used at Sacidava Fortress, providing valuable insights into the ancient settlement.*

Keywords: *Sacidava; materials; stone; Roman empire;*

Introduction

Sacidava Fortress is a Geto-Dacian settlement and Roman castrum, from the Roman era (Trajan era)[6]. It is located in Dobrudja region, Romania, on a hill on the right bank of the Danube - the Muzait point, located at about 5 km north-east from Dunăreni village (Figure 1). Between the villages of Rașova and Dunareni, on a high hill on the right bank of the Danube, at the Musait point there is a fortification of the Scythian limes, with several development phases, having an uninterrupted evolution since the beginning of the 2nd century AD until the first quarter of the 7th century BC[7]. The Roman fort was built on the territory of a former Dacian fortress, which seems to have been their capital. Roles, their leader, was an ally of Octavian Augustus. The name Sacidava is known from ancient literary sources and has been confirmed by epigraphic discoveries. Discovered and identified by Vasile Parvan, the fort was insufficiently researched, until 1969.

The Roman Sacidava was part of the province of Scythia, at its southern limit, it belongs to the military complex (limes) built along the Danube since the 1st century AD. In the immediate vicinity of the Roman Sacidava there is a Getic settlement, located at about 200 m east of the Roman fortified settlement, and which has a continuity of habitation from the 4th century BC and

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[6] Constantin Scorpan. 1973. ″Săpăturile arheologice de la Sacidava″. *Pontica VI:* 267-331.
[7] Dario Camuffo. 1986. ″Deterioration Processes of Historical Monuments″. *Studies in Environmental Science 30:* 189-221.

until the end of the 1st century BC (the toponym Sacidava suggests that in that place or nearby there was a Getic settlement before the Roman conquest). The second complex, an Early Medieval settlement from the 9th – 10th centuries AD is located at about 600 m east of the Roman Sacidava.

Archaeological studies thus continued, with some interruptions, until 1980. In the last three years, little research has been made[8,9].

Figure 1. *The aerian photo of Sacidava fortress (photo from personal archive of authors)*

The quality of Roman mortars is special, due to their high durability) they survived two millennia) in very good conditions. This is the reason for what these mortars should be investigated. The building materials and the number of case studies in archaeological sites are increasing day by day[10],[11],[12],[13],[14]. However, no sufficient data about the Roman mortars are

[8] Rodica-Mariana Ion & Lorena Iancu & Ramona Grigorescu & Sorin Colesniuc & Verginica Schroder & Raluca Andreea Trandafir & Silviu Ionita & Anca Gheboianu & Sofia Slamnoiu-Teodorescu. 2023. ″Diagnosis, Photogrammetry and Conservation Treatment with Nanomaterials of Sacidava Fortress″. *Chemistry Proceedings* 13: 25.

[9] Shobhit Pathak & Abhishek Kumar Singh. 2020. ″Deterioration Processes of Historical Monuments″. *International Journal of Innovative Research in Science, Engineering and Technology (IJIRSET) 9(8):* 7520-7523.

[10] Patricia Adriano & Antonio Santos Silva & de Rosario Veiga & Jose Mirão & Antonio Candeias. 2009. ″Microscopic characterisation of old mortars from the Santa Maria Church in Évora″. *Materials Characterisation* 60(7):610–620.

[11] Giovanni Borsoi & Antonio Silva Santos & Priscila Fernanda Campos De Menezes & Antonio Candeias & Jose Mirão. 2019. ″Analytical characterization of ancient mortars from the archaeological roman site of Pisões (Beja, Portugal)″. *Construction and Building Materials* 204*:* 597-608.

[12] Domenico Miriello, & Donatella Barca & Andrea Bloise & Annamaria Ciarallo & Gino Crisci & Teresa De Rose & Caterina Gattuso & Flavia Gazineo & Mauro La Russa. 2010. ″Characterisation of archaeological mortars from Pompeii (Campania, Italy) and identification of construction phases by compositional data analysis″. *Journal of Archaeological Sciences* 37: 2207–2223.

[13] Stefano Columbu & Carla Lisci & Fabio Sitzia & Giulia Lorenzetti & Marco Lezzerini & Stefano Pagnotta & Simona Raneri & Stefano Legnaioli & Vincenzo Palleschi & Gianni Gallello & Benedetta Adembri. 2018. ″Mineralogical, petrographic and physical-mechanical study of Roman construction materials from the Maritime Theatre of Hadrian's Villa (Rome, Italy)″. *Measurement* 127: 264–276.

[14] Concetta Rispoli & Lorenzo Fedele & Claudia Di Benedetto & Renata Esposito & Sosio Graziano & Vincenza Guarino & Vincenzo Morra & Piergiulio Cappelletti. 2019. ″Characterization of building materials from the Anfiteatro Flavio (Pozzuoli, Southern Italy): A mineralogical and petrographic study″. *Italian Journal of Geosciences* 138: 103–115.

known. Many external facts (geological of raw materials, climate, intermediary conservation, etc.) should be taken into account[15].

The present work aims to approach through advanced techniques the investigation of stone materials from this archaeological site (composition by techniques such as XRF, FTIR, Raman, XRD, morphology of surfaces/layers: optical microscopy, stereomicroscopy), which served to identify the materials, components of the stone, and their degradation degree.

1. Experimental part

1.1. Materials

The study was conducted on several few mortar samples collected from the archaeological area tower A, the east gate (Sacidava fortress, plateau of Muzait hill). The samples were taken from 4 distinct points on different looking surfaces: compact or porous material, bonding material between pieces (mortar type).

1.2. Methods and equipments

The thermography measurements have been achieved with the Catepillar 61 camera, with FLIR analysis system, thermal camera serial (2524095). For interpretation, several humidity and temperature distribution diagrams were made.

The morphological aspects were examined under stereomicroscope (OPTIKA) and epifluorescence microscope (Optika B350). For microscopic observations, extemporaneous preparations were made, and specific stains were made with orange acridine. The epifluorescence analysis method makes it possible to identify *in vivo* the cytological status and to identify some of the released substances such as secondary metabolites, as evidenced by different emissions (green or red). Harvesting was carried out from the surfaces of the stones using a sterile swab (4 cm^2). 12 samples were taken from flat surfaces and with different different porosities. The samples were transported to sterile enclosures and inoculated directly into Sabouraud Dextrose Agar culture media (selective medium for fungi) and Nutrient Agar (selective medium for bacteria), solids located in petri dishes. The plates were placed in the incubator at 350C. Colonies were analyzed 24 hours and 48 hours after inoculation.

Also, for morphological investigations, the samples subjected to analysis by FESEM-FIB type workstation (field emission electron microscope for electronic and ion focused beam) Auriga model produced by Carl Zeiss SMT Germany through the secondary electron/ion detector (SESI) in the sample chamber for the topography/surface morphology of the analyzed samples.

The verification of the chemical composition was performed using the EDS probe (dispersive energy spectrum for characteristic X-radiation) produced by Oxford Instruments, UK - X-MaxN dispersive energy spectrometer model with Aztec acquisition and processing software integrated on FESEM-FIB Auriga workstation.

In this paper was used the entire spectrum of scanning electron microscopy analysis SEM-EDS from the morphology of the elements that constitute the analyzed surface to obtaining maps of distribution of the elements that constitute the surface.

[15] Rodica Mariana Ion & Marius Gheorghe Barbu & Andrei Gonciar & Gabriel Vasilievici & Anca Irina Gheboianu & Sofia Slamnoiu-Teodorescu & Madalina Elena David & Lorena Iancu & Ramona Marina Grigorescu. 2022. ″Multi-Analytical Investigation of Roman Frescoes from Rapoltu Mare (Romania)″. *Coatings* 12: 530.

2. Results and discussion

2.1. Evaluation of microclimatic factors on the archaeological substrate

The temperate-continental climate regime characteristic of Constanta County is influenced by the geographical position, situated between the Danube and the Black Sea, as well as by the physical and geographical peculiarities of the territory. The microclimate analysis was carried out by thermographic evaluation of the main analyzed pieces. The method allows the identification *in situ* of temperature and humidity variations, physico-chemical parameters that allow understanding the specific distribution and variation of biota[16].

Thermography allowed to highlight the fact that the presence of crusty lichens makes the covered surfaces register much higher temperature values than the support stone[17,18], without lichens, at the measured humidity of about 50%, Figure 2.

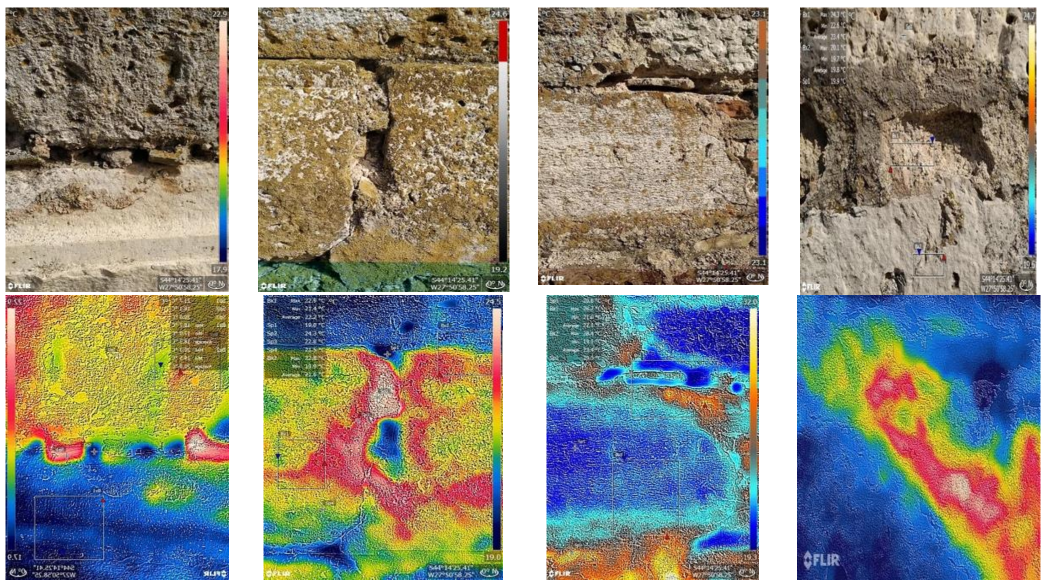

Figure 2. *Thermographic images of the investigated samples (experimental data obtained by the authors)*

2.2. Stereomicroscopy and SEM-EDS analysis

The historical mortar samples (samples TB) were characterized as lime mortars designed with a ground brick material (brick fragments of about 1 mm granule sizes). The results of the stereomicroscope analysis of the historical mortar sample indicate the kind of aggregates, as well as lime nodules, multistratigraphy, intrusions (Figure 3), as other authors already reported[19],[20].

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[16] Cristiana Radulescu & Claudia Stihi & Rodica Mariana Ion & Ioana Dulama & Geanina Stanescu & Raluca Stirbescu & Sofia Teodorescu & Valentin Gurgu & Dorin Let & Liviu Olteanu & Nicolae Stirbescu & Ioan Alin Bucurica & Radu Olteanu & Cristina Nicolescu. 2019. ″Seasonal Variability in the Composition of Particulate Matter and the Microclimate in Cultural Heritage Areas″. *Atmosphere* 10(10): 1-17.

[17] Dimitri Gavrilov & Roman Maev & Darryl Almond. 2014. ″A review of imaging methods in analysis of works of art. Thermographic imaging method in art analysis″. *Canadian Journal of Physics* 92: 341–364.

[18] Vasiliki Dritsa & Noemi Orazi & Yuan Yao & Stefano Paoloni & Maria Koui & Stefano Sfarra. 2022. ″Thermographic Imaging in Cultural Heritage: A Short Review″. *Sensors* 22: 9076.

[19] Duygu Ergenç & Rafael Fort. 2019. ″Multi-technical characterization of Roman mortars from Complutum, Spain″. *Measurement* 147: 106876.

[20] Sara Pavía & Susana Caro. 2008. ″An investigation of Roman mortar technology through the petrographic analysis of archaeological material″. *Construction and Building Materials* 22 (8): 1807-1811.

Old mortars exhibit various pathology symptoms like cracks, crumbling, and erosion after long exposure. To understand material aging and degradation over time, studying microstructure changes and measuring the area of damages in the form of micro voids is crucial. SEM analysis can depict loose microstructures in damaged materials and uniform and dense microstructures in durable ones (Figure 4). The damage caused by salts in porous materials is a common occurrence, leading to issues like cracks, scaling, efflorescence, crypto-florescence, pulverization, and material loss. Despite enduring wetting-drying cycles and extreme conditions, old mortars demonstrate remarkable durability and longevity. The crystallization of salts occurs when their internal pressures surpass the material strength, as indicated by research findings[21],[22],[23]. The analysis of mortars using SEM and EDS techniques revealed the presence of organic fibers along with minerals such as calcite, quartz, and muscovite in Ottoman mortars. The research team further identified hydration-dehydration products through morphological examination and microanalysis, noting a moderately uniform distribution of porosity that was linked to the homogenous distribution of hydration products[24]. Also, the presence of different elements, idenmtified by EDS (Figures 4, 5), are responsible for the high durability of these mortars[25].

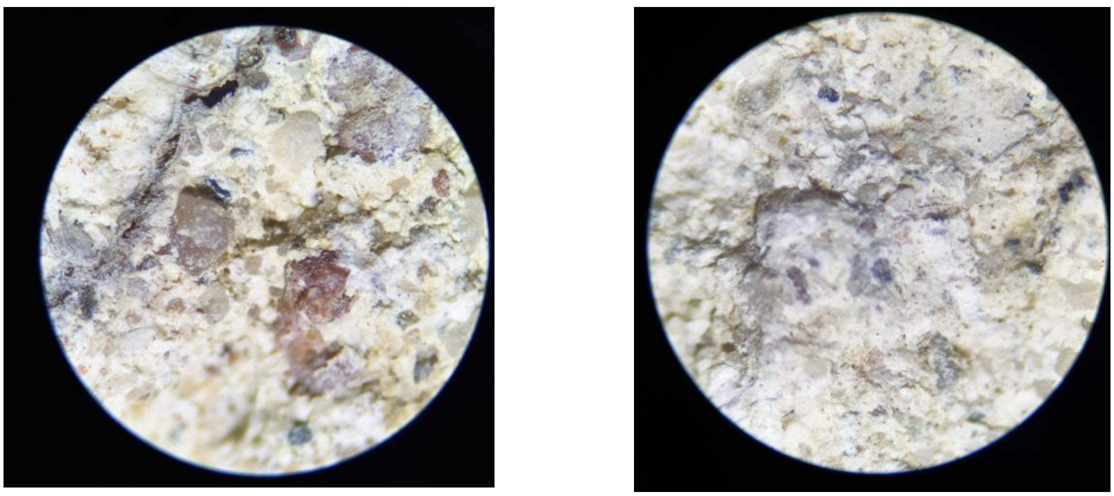

Figure 3. *Stereomicroscopy images of Roman mortar (experimental data obtained by the authors)*

[21] Francesca Gambino & Glarey Annie & Roberto Cossio & Lorenzo Appolonoa & Anna d'Atri & Alessandro Borghi. 2022. ″SEM-EDS characterization of historic mortar as a tool in archaeometric study: an updated analytical protocol tested on the Roman theatre of Aosta (NW Italy)″. *Archaeology and Anthropology Science* 14: 179.

[22] John Asiedu Larbi. 2004. ″Microscopy applied to the diagnosis of thedeterioration of brick masonry″. *Construction and Building Materials* 18: 299–307.

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[24] Hanifi Binici & Joselito Arocena & Selim Kapur & Orhan Aksogan & Hasan Kaplan. 2010. ″Investigation of the physico-chemical and microscopic properties of ottoman mortars from Erzurum (Turkey)″. *Construction and Building Materials* 24: 1995–2002.

[25] James Papike & James Karner & Charles Shearer. 2005. ″Comparative planetary mineralogy: Valence state partitioning of Cr, Fe, Ti, and V among crystallographic sites in olivine, pyroxene and spinel from planetary basalts″. *American Mineralogy* 90: 277–290.

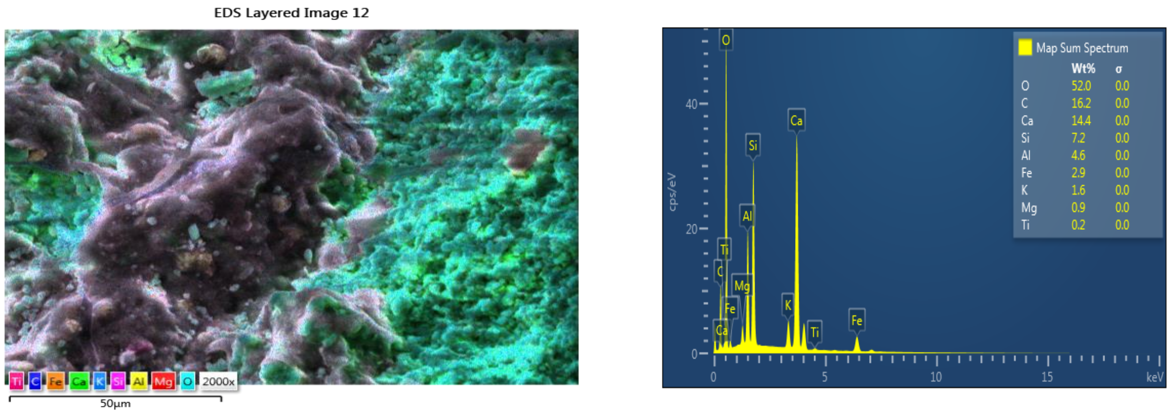

Figure 4. *SEM image (left) and EDS composition of investigated mortars (experimental data obtained by the authors)*

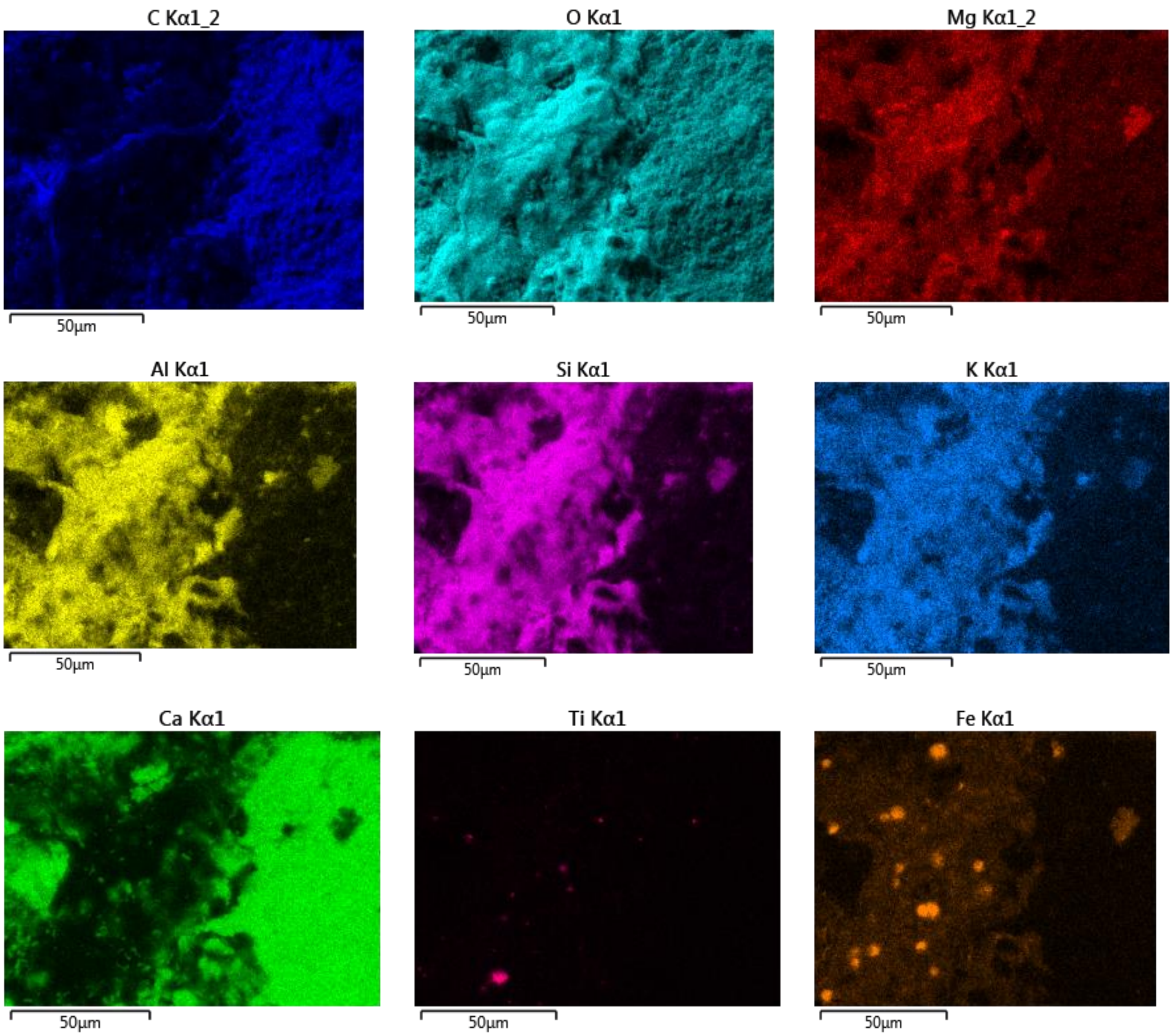

Figure 5. *The compositional map of the investigated mortar (experimental data obtained by the authors)*

2.3. Microbiological characterisation of samples

Each component was analysed for the samples taken from each relevant area, with macroscopic data corroborated by microscopic data. The analysis of the physical details of the analyzed fragments was performed under a stereomicroscope (magnification of 20x or 40x), and the details of colonization with microorganisms were analyzed microscopically and by the technique of isolation of microorganisms on artificial media, selective for bacteria and fungi. For identification, morphological features of colonies were analyzed and microscopic preparations were made.

All investigated samples have as a common element the extension of surface colonization of approximately 80-90% of the surface of the pieces. There is a low diversity of lichens on the assessed areas. Macroscopically, crusty, folious lichens and mosses are distinguished. Lichens with crusty thallus form compact elements on the surface of the stone substrate. The degree of microcolony coverage is high on intensely uneven calcareous structures. The structures of the microscopically analyzed rocks revealed the thallets penetrated into the substrate and the occurrence of degradation by fragmentation. On the surfaces of the talons can be highlighted apothecia-type formations, organs of fruiting. Mosses form isolated colonies, especially on sectors with higher humidity. Microscopically, the analysis allows the identification of epilytic components with visible biological activity, the external area (cuticle) being represented by autotrophic activity, with red emission, and the internal one (medulla), having green-yellow emission, includes fungal hyphae (branched) [26],[27].

The samples collected from the analyzed substrate are characterized by very specific biological activity induced by crusty lichens. Lichens have been identified that form compact plectenchymatic structures formed by hyphae arranged in the medulla (Figure 6). The polarization of structures is evident, the algal cortex being peripherally active (Figure 6 b,c), and spheroid structures (metabolites) are highlighted in the subcortical area (Figure 6 b,d).

Lichens are a complex group of symbionts, slow-growing thallus organisms, capable of developing metabolic processes under special conditions, such as high temperature and low humidity. They are unique symbionts between fungi and algae and are distinguished in the plant world by their specific secondary metabolites with phenolic structures. In response to biological and abiotic stress, secondary metabolites are either extracellular, as crystals on the surface of fungal hyphae, or intracellular (Figure 6 b, d).

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[26] Agnes Mihajlovski & Damien Seyer & Hayette Benamara & Faisl Bousta & Patrick Di Martino. 2015. ″An overview of techniques for the characterization and quantification of microbial colonization on stone monuments″. *Annals of Microbiology* 65: 1243–1255.

[27] Jie Chen & Hans-Peter Blume & Lothar Beyer. 2000. ″Weathering of rocks induced by lichen colonization – A review″. *CATENA* 39: 121–144.

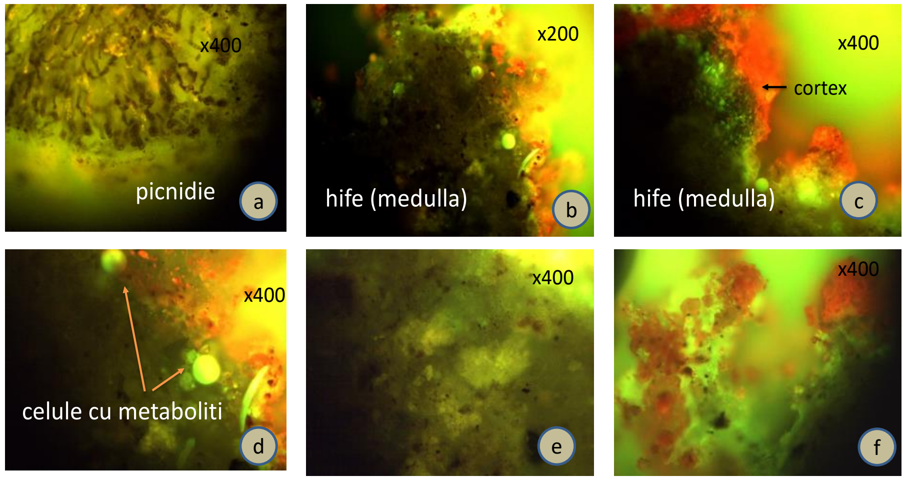

Figure 6 *Epilytic lichens – microscopic details (epifluorescence; orange acridine staining) morphological (b,c,e,f) and reproductive (a) structures, accumulations of metabolites (b,d) (experimental data obtained by the authors)*

2.4. Collection and processing of images and technical specifications taken by drone

The image collection and processing system is based on the Mavic 2 Enterprise professional drone for aerial image acquisition and on a series of application programs for analyzing and processing images captured from archaeological sites.

Technical specifications of image processing software components. Application programs do the following: Image reading functions, Dimensional adjustment functions, Chromatic analysis functions, Morphological analysis functions, Statistical analysis functions of image elements (pixels), Image classification algorithms.

2.5. Preliminary study of the aerial photogrammetry system in the investigated archaeological site.

In photogrammetry operations, the drone captures a large number of high-resolution photos on the objective area of interest (Figure 7). These images overlap so that the same point on the ground is visible in several photographs and from different points of view. Photogrammetry uses these multiple viewpoints in images to generate a 3D map[28,29].

[28] Galina Trebeleva & Konstantin Glazov & Andrei Kizilov & Suram Sakania & Vladlen Yurkov & Gleb Iurkov. 2021. ″Roman Fortress Pitiunt: 3D-Reconstruction of the Monument Based on the Materials of Archaeological Research and Geological Paleoreconstructions″. *Applied Sciences* 11: 481.

[29] Gurcan Büyüksalih & Kan Tuna & Gozde Enc Özkan & Muge Meriç & Lale Isın & Thomas Kersten. 2020. ″Preserving the knowledge of the past through virtual visits: From 3D Laser scanning to virtual reality visualisation at the Istanbul Çatalca İnceğiz caves″. *Journal of Photogrammetry and Remote Sensing Geoinformation Science* 88: 133–146.

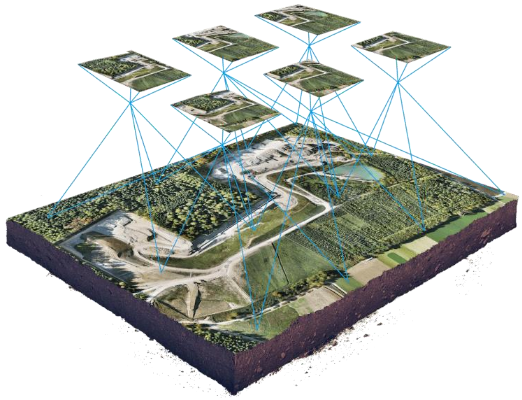

Figure 7 *The principle of aerial photogrammetry (experimental data obtained by the authors)*

At the archaeological objective Sacidava Fortress, the drone was tested in manually controlled flight to capture images with the size 4000x3000 pixels. The local weather conditions were: wind speed 3.5 m/s with gusts up to 5m/s, air temperature 11^0C, air humidity 56%, very good visibility, sunny. The areas with visible ruins were scanned, panoramic video sequences of larger areas of the fortress perimeter were captured, including photo and video with the areas of recent archaelogical excavations (Figure 8).

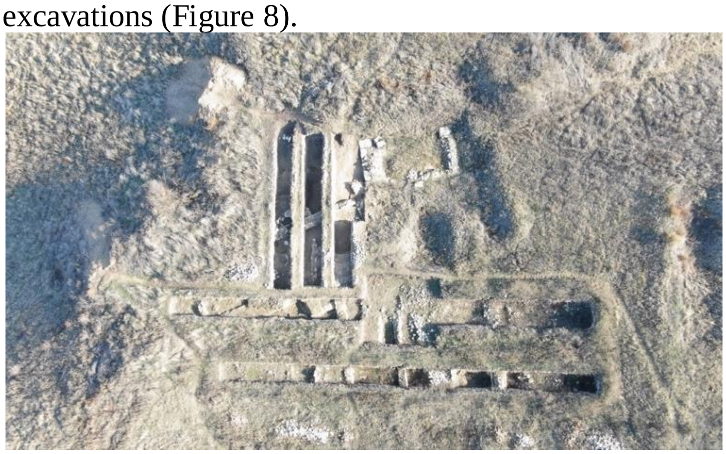

Figure 8 *Photo of recent excavations (experimental data obtained by the authors)*

3. Conclusions

The paper deals with the Roman mortars from Sacidava fortress and put into evidence the presence of carbonate phases, bound water, and hydraulic compounds such as silicate and aluminate hydrates. Thermographic images, stereomicroscopy images and SEM-EDS results have been correlated and discussed. Besides this, the microbiological analysis completed the obtained analytical data. This detailed study sheds light on the historical Roman mortar significance and construction methods used at Sacidava Fortress, providing valuable insights into the ancient settlement.

Acknowledgements

This work was supported by a grant of the Ministry of Research, Innovation and Digitization, CCCDI - UEFISCDI, project number PN-III-P2-2.1-PED-2021-3885, within PNCDI III.

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