The Scientific Bulletin of VALAHIA University
MATERIALS and MECHANICS –Vol. 20 , No. 22

DOI 10.2478/bsmm-2024-0002

3D RECONSTRUCTION AND NUCLEAR ANALYTICAL INVESTIGATIONS OF TROPAEUM TRAIANI MONUMENT, ADAMCLISI

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Abstract: The paper aims to conduct a novel investigation of ceramic samples from the Trophaeum Traiani Monument in Romania using various nuclear analytical techniques such as WDXRF, XRD, neutron diffraction, and neutron tomography, which will be complemented by FTIR and Raman spectroscopy data. The experimental data gathered revealed the mineral and chemical composition of the object's surface, identifying quartz, hematite, calcite, and graphite. The employment of non-destructive techniques with high penetration capabilities is adequate in examining the spatial distribution and phase composition of the samples, providing valuable insights into their composition and structure without altering their integrity.

Keywords: *Adamclisi, analytical investigations, nuclear techniques, neutron diffraction, neutron tomography*

1. INTRODUCTION

The current trend in scientific research concerning cultural heritage is focused toward to non-invasive methods and digital technologies. These approaches allow for the preservation and study of historical artefacts and monuments without causing damage, offering new insights and perspectives through advanced technological tools and processes [1].

The Tropaeum Traiani stands as a historic monument commemorating Roman Emperor Trajan's triumph over the Dacians, in the winter of 101-102. Built in 109 on the site of the Battle of Adamclisi in modern-day Romania, it honors the sacrifices of the legionaries and servicemen who fought and died in the conflict "fighting for the Republic". The monument's construction served as a symbol of Roman power and conquest in the region.

The Tropaeum Traiani, discovered in Romania in the early 20th century, holds significant historical value is recognized as a very important ancient Roman monuments in the region, Figure 1.

These ruins in the town of Adamclisi, in the Dobrogea region, mark the site of battles between Romans and Dacians in 101/102 A.D. The triumphal monument at Adamclisi celebrates Emperor Trajan's victory over Dacia and is an architectural masterpiece attributed to the renowned architect Apolodor from Damascus.

Figure 1. Adamclisi monument (personal photo)

Despite facing degradation over time due to factors like earthquakes and attacks, the circular monument remains an impressive symbol of ancient Roman conquest and military prowess [2].

The term "trophy" has a range of meanings, including a symbol of victory such as defeated armour displayed on a tree trunk, a monument made of stone or marble commemorating military triumphs, a sculpture or painting depicting a monument, or spoils of war taken from the enemy. In Latin and Greek, "tropaeum" and "tropaion" refer to triumphal monuments built to celebrate military victories in ancient Greece and Rome, dedicated to consuls, military leaders, and naval conquests, both within and outside the territories of these ancient civilizations.

The discovery and reconstruction of the ancient Roman ruins of the triumphal monument and fortress of Tropaeum Traiani in Adamclisi, Constanta County is of immense historical importance. The process of rebuilding the monument used a combination of historical accuracy and modern technology, using a metal structure covered with stone pieces from the original quarry [3,4].

The research on the stone weathering, due to various climatic conditions and pollutants, has been extensive and varied. Various studies have examined the effects of temperature, relative humidity, and pollutants, as well as the formation of salt deposits, accretions, and weathered surfaces on stone sculptures and buildings. Additionally, the formation of calcium oxalate as a weathering product from the action of lichens on stone has been investigated in terms of its stability and potential for protecting underlying stone. The studies mentioned in the literature have played a crucial role in enhancing our understanding of how to effectively preserve and conserve stone structures that are vulnerable to weathering processes. [5,6].

In recent years, neutron radiography and tomography have emerged as powerful tools for analyzing the structures of materials under different conditions of relative humidity and temperature. These techniques allow for the examination of object samples ranging from a few millimeters to hundreds of centimeters in size, with a high resolution of around hundred microns. The utilization of neutron imaging in the field of cultural heritage provides valuable insights into the composition and preservation of historical artifacts, offering researchers a non-invasive method to study and protect these objects [7].

The primary objective of the paper is to conduct a complex analysis of the samples, drawing from the findings of previous joint research programs conducted by our group (2020).

In addition to nuclear techniques, the study will also present spectral techniques like Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy to assess the chemical composition of selected artifacts.

2. MATERIALS AND METHODS

2.1 Samples

The monument fragments of irregular shape with some cavities has dimensions of approximately 13 × 10 × 8 mm (Fig. 2). The cavities are covered with a layer of brownish patina called "scialbature". The sample P10A was only for microscopic visualization and for neutron tomography.
The used samples collected from Adamclisi monument, are shown in Fig. 2.

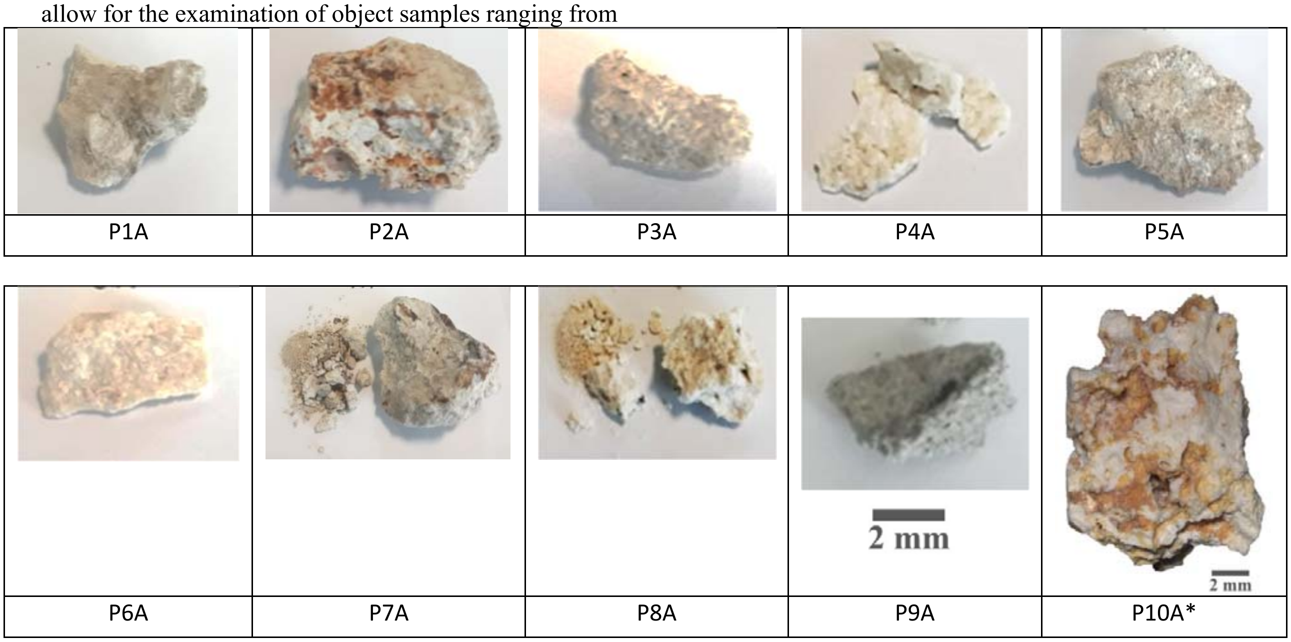

Figure 2. Photo of the front and back side of the studied sample. A scale bar is shown.
*** The sample P10A was used only for nuclear investigations**

2.2 Experimental methods

Images of the studied fragment of the monument were captured using a Leica M165 microscope with video camera setup, providing a maximum magnification of up to 8x, for a detailed visualization and analysis of the samples, enabling rto explore the structure and features of the monument for in-depth examination and documentation.

The Ultima IV diffractometer (Rigaku in Woodlands, TX, USA), utilizes Cu Kα radiation for its analytical capabilities. (with a wavelength of 1.54 Å), conducted

X-ray diffraction (XRD) measurements using Bragg–Brentano beam geometry within the range of 2θ = 10° to 100°. The scan rate was set at 1 degree per minute for the diffraction patterns identification for all the samples.

The Supermini200 spectrometer, manufactured by Rigaku in Woodlands, TX, USA, utilized a Pd X-ray tube with a power of 200 W, incorporating two detectors and three analyzer crystals. This instrument offered a detection limit ranging from 0.1 to 1 ppm and achieved precision up to 0.5 percent, enabling the execution of wavelength dispersion X-ray fluorescence (WDXRF) analysis for the sample.

The phase composition analysis of the sample was carried out through neutron diffraction experiments utilizing the DN-6 neutron diffractometer at the IBR-2 high-flux pulsed reactor in Dubna, Russia. Neutron powder diffraction patterns were obtained at a scattering angle of 2θ = 90°, with each pattern requiring 30 minutes for measurement. Analysis of the diffraction patterns was performed using the FULLPROF software in profile matching mode to understand the phase characteristics of the sample [8].

Raman spectra were obtained at ordinary temperature using a LabRAM HR spectrometer with a He–Ne laser emitting at 633 nm, a 1800 grating, 100 µm confocal hole, and a × 50 objective. Multiple spectra were recorded from various sample surface points, with a typical acquisition time of 12 minutes and a range of 100 to 1800 cm^{-1}. The Raman signatures of crystalline phases were identified through comparison with information from online databases and literature references [9].

For recording Fourier-transformed infrared spectra (FTIR), a Vertex 80 spectrometer (Bruker Optik GMBH, Ettlingen, Germany), has been used.

The neutron tomography experiments at the IBR-2 high-flux pulsed reactor utilized a detection system with a high sensitivity camera for collecting radiography images and employed a rotation step of 0.5°with 360 radiography projections taken over a period of 6 hours, with a projection of 20 s aa exposure time. Image correction and normalization processes were carried out using ImageJ software, with an accurate and consistent adjustments of the image data, while the tomographic reconstruction was achieved through through the SYRMEP Tomo Project (STP) software, resulting in a vast dataset of 3D voxels with a size of 52 × 52 × 52 µm.

With an adequate spatial resolution of the equipment, a minimum item size of 135 µm with the 3D voxel data could be resolved. Neutron attenuation coefficients in the sample volume, was further analyzed using VGStudio MAX 2.2 software of Volume Graphics (Heidelberg, Germany) for visualization and analysis of reconstructed 3D data [10-12].

3. RESULTS AND DISCUSSION

3.1. XRD/ WDXRF and neutron diffraction results

From the investigated monument have been collected 9 samples (from the surface to the interior in depht) and analyzed as it is shown in Table 1.

Table 1. Chemical composition of Adamclisi samples by XRF

Oxid Comp.	Concentration (%)							
	2A	3A	4A	5A	6A	7A	8A	9A
CO_2	33.5048	32.4571	42.2175	37.8113	43.981	32.0718	43.9505	21.8447
Na_2O	0.1886	0.0907	0.0665	0.0579	-	0.6323	-	0.3893
MgO	1.0266	0.4694	0.3118	0.3498	0.2758	1.2722	0.2565	0.7437
Al_2O_3	4.5837	1.5653	0.2795	0.8555	0.1384	3.2905	0.0953	4.1338
SiO_2	21.2605	26.4803	1.809	15.7059	1.0212	22.1998	0.2794	49.5692
P_2O_5	0.0957	0.0801	0.0621	0.0761	0.066	0.1125	0.0879	0.0554
SO_3	0.2345	0.0505	0.0406	0.0421	0.0175	0.1097	0.0284	1.3948
Cl	0.045	-	-	0.0436	-	0.2883	-	0.0286
K_2O	0.9579	0.2997	0.0506	0.1532	0.0316	0.8635	0.0097	0.9196
CaO	35.526	37.8181	55.0203	44.4505	54.3685	37.4064	55.2129	18.7811
Fe_2O_3	2.2168	0.676	0.1169	0.4393	0.0794	1.3691	0.0555	1.823
SrO	0.0217	0.0127	0.0252	0.0147	0.0206	0.0369	0.00431	0.0292

By analyzing this table, could be concluded that the composition of this monument is not homogeneous, exhibiting different oxides concentrations in different places. For example, in the samples 4A, 6A and 8A, SiO_2 is missing, while very high concentrations of CaO are registered. The explanation could be the different sourse of the raw materials from Dobrogea, or previous interventions on this monuments by using limestone [13,14]. Analyzing these results, the following conclusions can be drawn:
1. The samples were taken from the surface to the interior of the monument;
2. Calcite concentration decreases from the surface to the monument depth;
3. The concentration of graphite decreases from the surface in-depth, proof of the presence of ash resulting from the fire that this monument went through;
4. The concentration of silica increases from the surface to the interior, simultaneously with the concentrations of other metals/minerals (Ti, Zr, Fe, Sr. Ag), which denotes the decision of the Romanian builders to create a lasting monument.
Similar results have been obtained by XRD measurements, Figure 2.

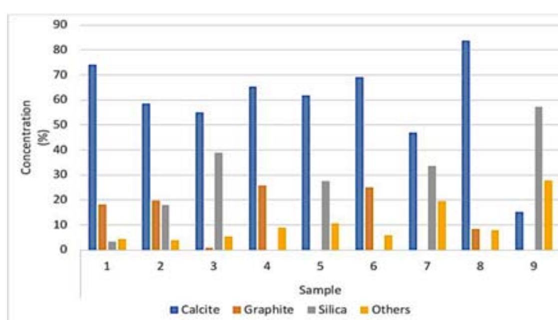

Figure 2. XRD distribution for all minerals

All these results are in good agreement with some geologic data reported in the literature, which put into evidence the specific earth from Adamclisi (diatomaceous earth found in the geological samples that alternates with bentonitic clays and limestones). This diatomite can exhibit specific characteristics: very fine-grained, soft, chalk-like, friable, white-yellow color [15].

The neutron diffraction patterns depicted in Figure 3 display typical peaks corresponding to the predominant phase identified in the monument fragment: calcite CaCO3. By analyzing these diffraction peaks, the unit cell parameters of calcite can be accurately calculated, providing valuable insight into the crystal structure and composition of the material under investigation. The calculated unit cell parameters of calcite are a = 4.987(1) Å, c = 17.060(9) Å. The basis of the intact part of the sample is still marble. The phases of calcium oxalates were not found, which indicates a small thickness of their layer. This is due to the fact that whewellite and weddellite formed on the surface of calcite under the action of oxalic acid have an extremely loose and porous structure [16] and are washed out over time.

Similar results with neutron diffraction could be obtained with x-ray diffraction (phase composition). The neutron radiography and tomography can provide details about the spatial distribution of internal components. Additionally, neutron interactions with matter offer a high sensitivity to light elements and good contrast between close elements. These features make neutron-based methods valuable tools for materials analysis and imaging applications.

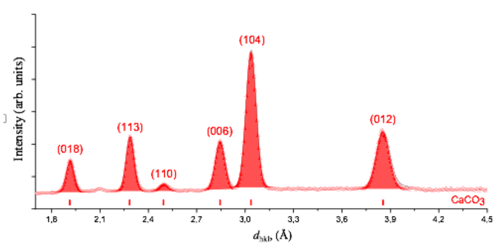

Figure 3. Neutron diffraction patterns of the monument fragment sample. The experimental points and calculated profile are shown. The Miller indices of the phase are labeled. Ticks below represent calculated positions of the Bragg peaks of the calcite phase. The corresponding diffraction peaks are marked

3.2 Optical microscopy

The conventional optical microscopy allows identification of the phases that make up the material of the fragment of the monument. The surface of this fragment is represented by a white crystalline substance that can be identified as calcite (Fig. 4). Large and branched cavities are also visible. The characteristic appearance (large rounded grains) of these cavities indicates the presence of a calcium oxalate $CaC_2O_4 \cdot (H_2O)$ layer in them. The brownish tint of the cavities may indicate the presence of iron oxides, which is also very typical for such samples.

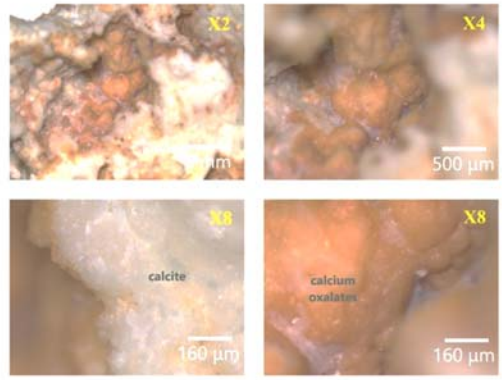

Figure 4. Microphotographies of a fragment of the monument. The magnification levels and corresponding scales for each image are marked. The tentative assignment of several fractions: calcite ($CaCO_3$), calcium oxalates ($CaC_2O_4 \cdot H_2O$, $CaC_2O_4 \cdot 2H_2O$), are shown

3.3 Neutron tomography

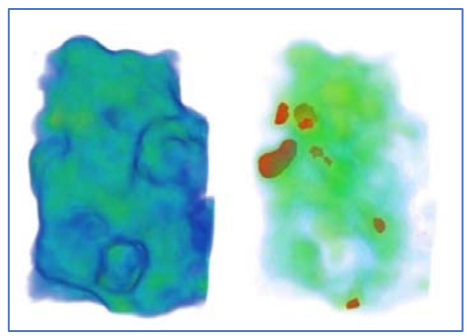

Figure 5. Reconstructed 3D model of a fragment of the monument

The spatial distribution of cavities and density inhomogeneities were analyzed by neutron tomography. Figure 5 shows a three-dimensional model of the sample reconstructed from tomographic data. Complex relief and all cavities are clearly visible on the model. In this case, a 3D virtual volume of the whole fragment of 2778688 voxels, was identified, which corresponds to the volume of 391 mm^3.

For a neutron beam (wavelength of ~ 2 Å), the neutron attenuation coefficient is in the range of Σ=0.282–0.372 cm^{-1}. This uncertainty is associated with the different density of marble areas, as well as the presence of microcracks and micropores. The volume of a fragment of the monument, which can be associated with low-density material, is 114 mm^3 or 29% (blue area in Figure 6). These areas are partially destroyed by oxalic acid, as they have a reduced density [16]. The total volume of large cavities, in turn, is ~10% of the sample. Areas of increased absorption of the neutron beam (red in Figure 5) were also found, which indicate the presence of other minerals or organic compounds typical to soft stone

[17]. The volume of these impurities is 23 mm^3 or 6% of the sample.

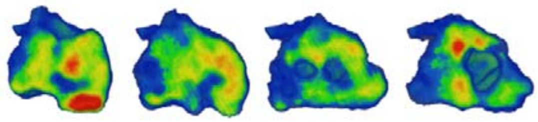

Figure 6. The visualization presents multiple virtual slices of the 3D model, with a color gradient ranging from blue to red indicating the varying levels of neutron absorption. The rainbow-like coloring scheme helps to visually represent the absorption degree across different sections of the model, allowing for a clearer interpretation of the neutron interaction within the material. The yellow-green areas could be attributed to marble with normal density, and the blue areas to marble with microcracks and micropores. Several red areas correspond to small amounts of other minerals or organic compounds. On slices it can be seen that cavities have a larger volume than is visible on the surface of the sample

3.4 Raman spectroscopy

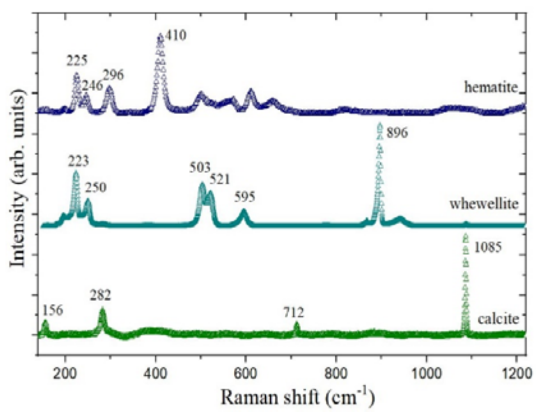

Figure 7. Raman spectra obtained from the monument fragment. The Raman wavenumbers of the most intense Raman peaks are indicated

Using Raman spectroscopy, calcite was found corresponding to the base material (marble) of the studied fragment of the monument. The additional phases detected indicate the presence of calcium oxalates and iron oxides.

3.5 FTIR data

Vibrational spectroscopy techniques, such as infrared and Raman spectroscopy, have been extensively utilized in chemicals characterization due to their ability to provide information on molecular structure and intermolecular interactions through vibration band frequencies and intensities.

The calcium oxalates could adopt three forms: calcium oxalate monohydrate (COM) as a prevalent mineral in nature, primarily formed through the reaction between oxalic acid and calcium ions, calcium dihydrate (COD) and calcium trihydrate (COT) which could be generated only in some specific conditions [26].

The infrared spectrum of calcium oxalate monohydrate (COM) in the region of 3700–2800 cm^{-1} contains O-H stretching vibration bands that overlap, indicative of crystallographic water molecules presents in the structure. Conversely, the bands representing fundamental vibrations of the oxalate anion are situated in the range of 1800–700 cm^{-1}, providing important information about the molecular structure and composition of COM, Figure 8. The assignment of fundamental vibrations of the calcium oxalate anion at 1805, 1460, 1409, 1090, 725 cm^{-1}, respectively, has been identified, confirming the Raman data. Also, sulphate anion is identified in FTIR spectra (1690 cm^{-1}), as a patina generated due to stone weathering.

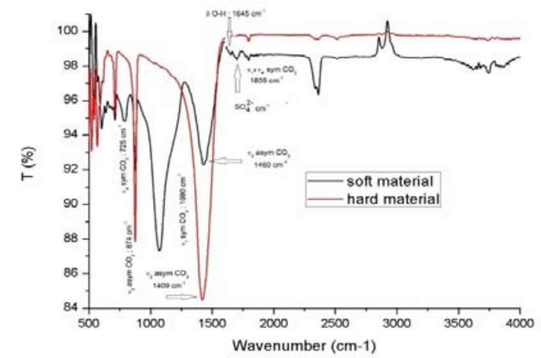

Figure 8. FTIR spectra of stone material from Adamclisi monument

4. CONCLUSIONS

Experimental data obtained from the surface of an object can be verified using nuclear techniques like wavelength dispersive X-ray fluorescence (WDXRF), X-ray diffraction (XRD), neutron diffraction, and neutron tomography, coupled with Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy. Neutron tomography enables the exploration of phase distribution within the object, while X-ray and neutron diffraction techniques assist in identifying the phase composition of the analyzed samples, providing a comprehensive understanding of the chemical and mineral composition of the material.

The results put into evidence the composition of the Trophaeum Traiani samples: calcite, hematite, and quartz, with small area of mono and di-hydrated calcium oxalates formed on the surface of calcite responsible for the porous structure. By using the non-destructive techniques with a high penetration rate into bulk objects appears to be an appropriate approach in this scenario. Such methods can help in examining the internal structure and composition of the object without causing damage, allowing for a comprehensive analysis while preserving the integrity of the material being studied.

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