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INFRARED SPECTROSCOPIC INVESTIGATIONS OF THE NORTHERN MOLE OF PORTUS, THE ANCIENT HARBOUR OF ROME. INSIGHTS FOR STRATIGRAPHY AND PROVENANCE OF RAW MATERIALS FOR CONSTRUCTION

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ABSTRACT

Despite numerous studies focusing on Ancient Roman concrete especially petro-chemical characterization and provenance investigations of aggregates and mortar, less is known about the raw materials used for the construction of harbour structures where concrete had not been used. Recent geoarchaeological investigations of the northern mole of the Claudius harbour (Imperial port of Rome, Portus), revealed ten meters of continuous stratigraphic succession of raw materials employed for the foundation of the mole structure where the water column at the time of construction was reaching more than 16 meters of depth. The material succession consists of volcanic tuffs of different quality and hardness, carbonate and quartz rich sand, fragments and boulders of magmatic rock and Roman sherds. Attenuated total reflectance infrared spectroscopy and principal component analysis have been applied on drilled core sediments and local reference outcrops including tuffs and pozzolana in the vicinity of Rome with the aim to: (i) characterize the mineral composition of samples and (ii) provide insights about the lithological provenance of raw materials. The results revealed that the majority of materials have a close spectra resemblance with "*Tufo Lionato*" and "*Pozzolane rosse*" except leucitite fragments occurring at the top of the harbour structure. By providing spectra similarities between the raw construction materials and the local outcrops, this study provides insights about the sources of materials and building strategies during the construction of the Imperial harbour of Rome and therefore constitute a starting point for further archaeological, geoarchaeological and restoration projects.

KEYWORDS: Infrared Spectroscopy, raw construction materials, provenance, ancient Roman harbour, mole

1. INTRODUCTION

Construction of the maritime harbour of Rome begun under the Emperor Claudius in the 1st century AD some 30 km southwest from Rome close to the mouth of the river Tiber (Figure 1). The rapid growth of Rome demanded increased food and material for the city, more than could be handled through the river port of Ostia Antica. The Claudian basin featured two long curved moles projecting into the sea and enclosing an area of around 220 hectares. However, by the 17th century the Imperial harbour was completely inland due to the infill sedimentation and coastal advancement (Giraudi, 2011). The terrigenous flux led to progressive landscape modifications and therefore a gap in our understanding of the harbour mole configuration (Giuliani 1996, Quilici 2017). However, significant advances in the understanding of the western extent of the harbour have been made, in particular during the constructions of Leonardo da Vinci Fiumicino International airport (Testaguzza, 1970). These findings have been supported by magnetometry (Keay et al., 2005), Ground Penetrating Radar (GPR) and Electrical Resistivity Tomography (Keay and Kay, 2018) surveys undertaken alongside the northern mole as well as by extensive drilling campaign to the west of the airport which aimed at intercepting the projectory of the mole structure (Morelli et al., 2011). Moreover, palaeoenvironmental and geoarchaeological investigations using drillings (Goiran et al., 2010) have allowed a better understanding of the configuration of harbour basins, moles, channel accesses (Goiran et al., 2008) as well as the depth of the basins (around 6-8 m below ancient sea-level, Goiran et al., 2009). Moreover, the obtained chronostratigraphy revealed the sedimentation rates and rhythm of basin infilling and therefore it is possible to indicate the type of vessels that had been able to access the quays, depending on their drought (Goiran et al., 2009). In addition, the Roman concrete used in the construction of the northern mole of Portus, has been investigated in the framework of the ROMACONS project using short cores (1.3 – 3 m depth, Oleson et al., 2004).

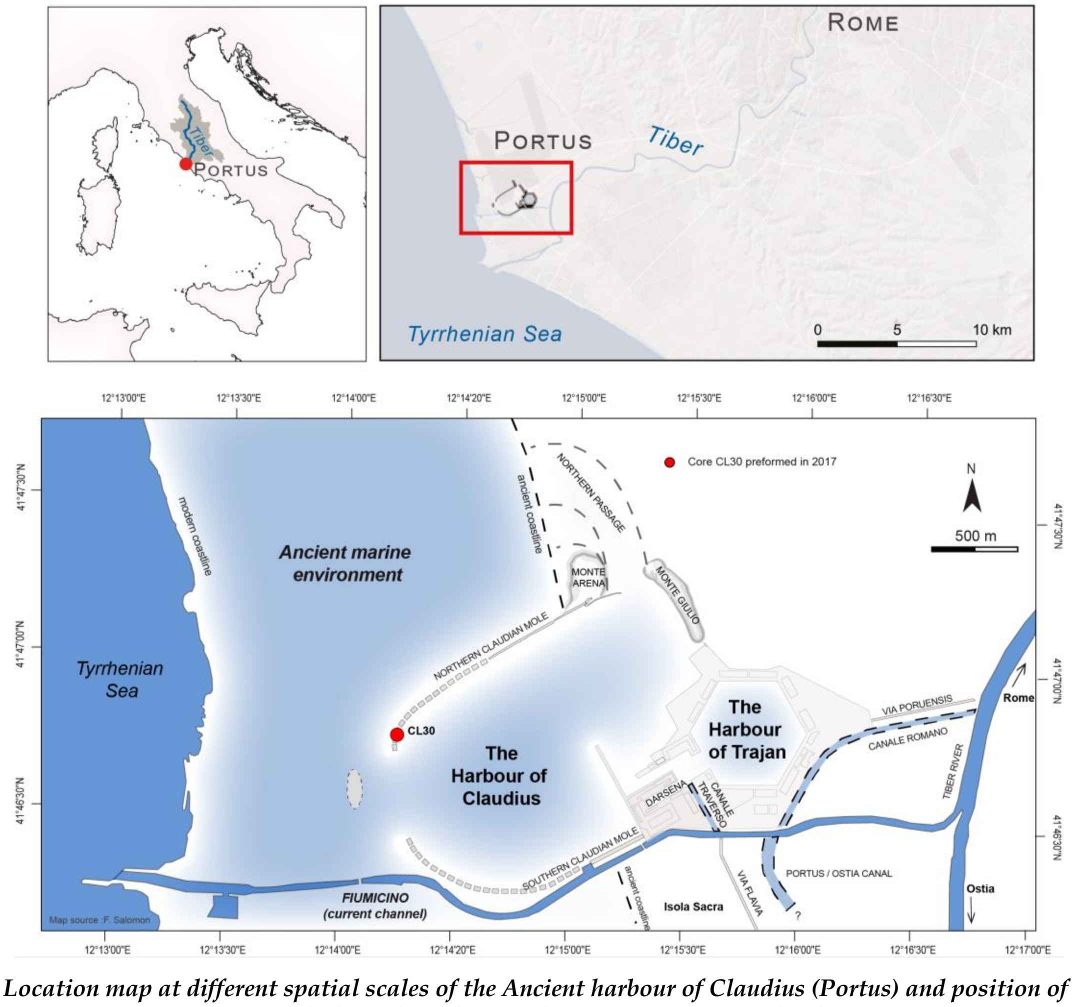

Figure 1: Location map at different spatial scales of the Ancient harbour of Claudius (Portus) and position of drill core CL30 into the buried northern mole (Map source: Ferreol Salomon).

Although significant progress has been made in the understanding of Portus as a harbour infrastructure, the lithological provenance of the raw material employed for the construction of the northern jetty is still to be done. Characterization and provenance of construction material are generally investigated using optical petrographic, mineralogical and element analyses (Theologitis et al., 2021; Miriello et al., 2010, 2011; Lezzerini et al., 2017; Marra et al., 2016, D'Ambrosio et al., 2015; Lancaster et al., 2011; Jackson et al., 2013; Hemeda, 2013). A detailed overview of the different techniques used has been proposed by Pezzolo et al. (2018). Sensitive to biochemical and inorganic compounds of sediments (Bertaux et al., 1998), minerals (Müller et al., 2014) and rocks (Chen et al., 2014), infrared spectroscopy (IR) is a rapid, non-destructive low-coast and efficient analytical method. It has been successfully applied in archaeometry for investigating Roman mortars (Al Sekhaneh et al., 2020; Ravisankar et al., 2013; Pezzolo et al., 2018; Kramar, 2011), ancient pottery (Manoharan et al., 2015; Velraj et al., 2009, 2012) and skeletons of Roman catacombs (Devièse et al., 2017). The principle of infrared spectroscopy relies on the interaction between the matter and the IR radiation. This latter induces molecular vibrations and rotational modifications in molecules leading to absorbance at specific wavenumbers of the IR electromagnetic spectrum. Therefore, the suite of wavenumbers which constitutes the IR spectrum is a unique fingerprint of the molecular composition of the sample (Farmer, 1974). Multivariate statistics, such as Principal Component Analysis (PCA) are commonly used (Poulenard et al., 2009) for spectra pattern recognition and natural clustering of the samples. Promising results obtained by PCA analysis demonstrate the potential of Mid-Infrared Spectroscopy (MIRS) to assess potential sediment sources of material employed by ancient Romans in construction activities (Pezzolo et al., 2018).

First attempts to characterize the raw materials from the norther mole of Portus with spectroscopic technics were performed in 2018 (Chapkanski et al., 2021c). However, using MIRS to investigate the possible lithological provenance of the raw material used in the construction of the northern mole of Portus is still to be tested. The hypotheses put forward here are the following: (i) samples of raw materials from the northern mole of Portus can be reliably characterized by their MIR spectra signature related to their mineralogical contents and (ii) the provenance of materials could be supposed based on reference samples from a mineralogical spectroscopy database and outcrops in the vicinity of Rome. To test these hypotheses, the study objective aims to apply MIRS-PCA model in order to: (i) identify the mineral compositions of samples and (ii) evaluate the mole material spectral similarities with the reference spectra.

2. MATERIAL AND METHODS

2.1. *Field drilling and sampling*

Investigations of the northern mole of the Claudian harbour began in 2017 by mechanical drilling (Core "CL30") into the buried harbour structures (Figure 1), previously identified by ERT (Keay and Kay, 2018) and by an earlier extensive campaign of coring (Morelli et al., 2011). A detailed stratigraphic description including colour of sediments, hand texture measurement, hardness as well as the presence of oxidation, shell, organic matter and plant remains was performed in the field and summarized in stratigraphic units.

In addition, outcrop surface samples have been collected from five sites in the vicinity of Rome (Figure 2) aiming to establish a local lithological reference dataset. The sampling strategy aimed at covering the potential lithological sources of the raw construction materials (RCM) as well as samples of Roman mortar. The obtained local reference collection comprises of 10 samples including mainly volcanic tuffs (*Tufo Giallo, Tufo del Palatino, Tufo Lionato and Tufo Rosso a Scorie nere*), Pozzolana (*Pozzolane Rosse* and Pozzolana used in the construction of the Roman Harbour of Frejus, France), Ignimbrite and mortars from the northern mole of Claudius harbour as well as from Frejus harbour.

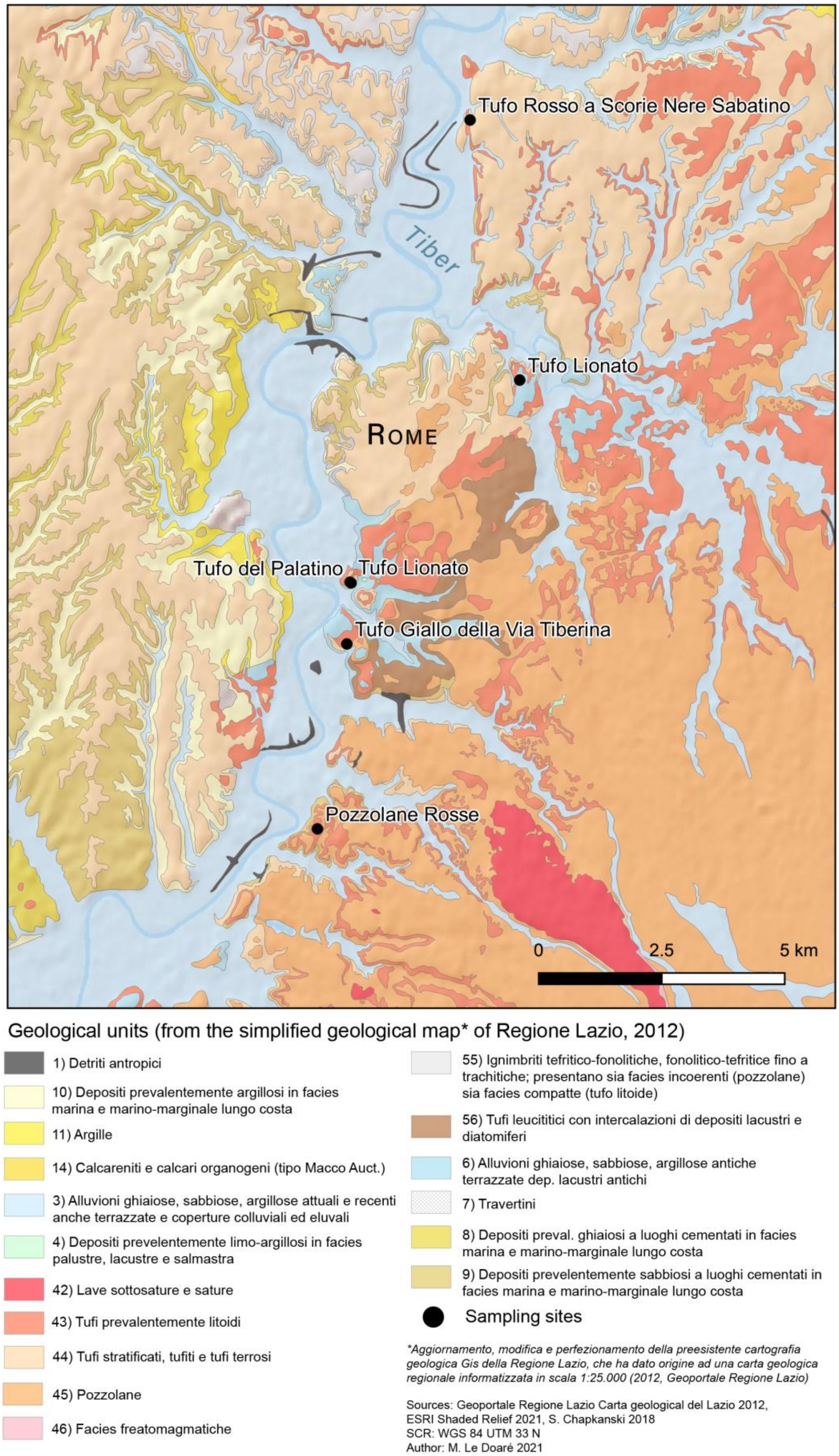

Figure 2: Simplified geological map in the vicinity of Rome indicating the local reference sampling sites. All samples present natural outcrop except the "Tufo Giallo della Via Tiberina" which is sample from the Servian wall.

2.2. *Laboratory work*

All samples (total of 82) including the mineralogical database, outcrop local references and borehole stratigraphic archives were dried in an oven at 50°C for seven days. Texture percentage of the borehole samples was established from 10 gr of sediments after wet sieving through a 0.063 mm and 2 mm stainless steel sieves. Furthermore, magnetic susceptibility measurements of 167 bulk samples were performed using a Bartington MS2 susceptibilimeter and MS2F probe (Oxon, UK). Samples for spectroscopic measurements including the reference dataset and borehole CL30 were ground to a fine homogenous powder using agate mortar and pestle and were additionally dried in an oven at 50°C for 24h. As the depth of infrared light penetration is low (from 0.5μm to 5 μm, Popov and Lavrent'ev, 1980), spectroscopic analyses were performed on powdered samples to ensure better infrared penetration into the sediment (Bertaux et al., 1998). Because this study focus on both raw materials for construction and natural sediments, the FTIR-ATR technic were used rather than a conventional X-Ray Powder Diffraction (Di Benedetto et al., 2018), because it is sensitive to both silicates, amorphous silica, carbonates and organic compounds. The mid-Infrared spectrum for each sample was obtained using a FT-IR Frontier Spectrometer with a KBr beam splitter, a universal attenuated total reflectance (UATR) sampling accessory and KRS5 - diamond crystal (PerkinElmer, Waltham, MA, USA). The powdered samples were scanned from 4000 to 350 cm^{-1} with a 2 cm^{-1} resolution. After the spectral acquisition, the data matrix was standardized and baselined using Standard Normal Variate (SNV) and Baseline pretreatments (Camo Unscrambler 10.3, Oslo, Norway). Principal component analysis (PCA) were performed with the same software. Supplementary technical data is detailed in Chapkanski et al., (2020a).

2.3. *Establishment of mineralogical spectra dataset*

A mineral spectroscopy database including pure minerals and rock outcrop references was established in order to improve the understanding of the association between the absorbance peaks at specific wavenumbers of the IR electromagnetic spectrum and the mineralogical and/or biochemical compounds contained in the investigated samples (Chapkanski et al., 2020c). The mid-Infrared spectrum for each mineral was obtained on powdered samples after several measurements in different ambient conditions i. e. temperature (18-26 °C) and humidity variations. Measurement repetitions guarantee the representativeness of the reference spectra. The sampling strategy for the mineralogical dataset aimed to establish a wide range collection covering potential minerals that could occur in unconsolidated sediments or rock outcrops. The obtained reference collection comprises 38 minerals including tectosilicates, inosilicates, phyllosilicates, nesosilicates, carbonates and nitrates as well as oxides/hydroxides, phosphates and sulphates.

3. RESULTS

3.1. *Stratigraphy of borehole CL30*

Core CL30 was drilled into the buried harbour structures and the stratigraphy presents three main units subdivided into a further three sub-units (Figure 3). The basal Unit 1 (- 22 to -18 m depth) is composed of grey silty clay fraction, for about 80% of the total weight of the sieved samples. The Magnetic Susceptibility (MS) indicates extremely low values. Unit B is massive (about 10 m depth) heterogeneous structure consisting of weathered crumbly coarse volcanic fragments at the basis (- 18 to - 15 m depth), hard pyroclastic aggregates in the middle of the unit (- 14.5 to – 9.5 m depth) and fragments of intrusive igneous rock overlaid by Roman sherds (- 9.5 to – 7.5 m depth, Figure 4). The MS indicates great variability with values ranging from 100 up to 2000 CGS. Unit C consists of interbedded sand, for about 60-70% of the total weight of the sieved samples. At the unit basis (Subunit C1), the interbedded sand contains broken shelly fragments (- 7.5 to - 6 m depth). Slightly weathered grey coarse sand occurs in the middle of the unit (Subunit C2). It is overlaid by yellowish interbedded slight silty sand. The MS indicates relatively low values where magnetic peaks seem to correlate with the presence of coarse sand and fine gravels.

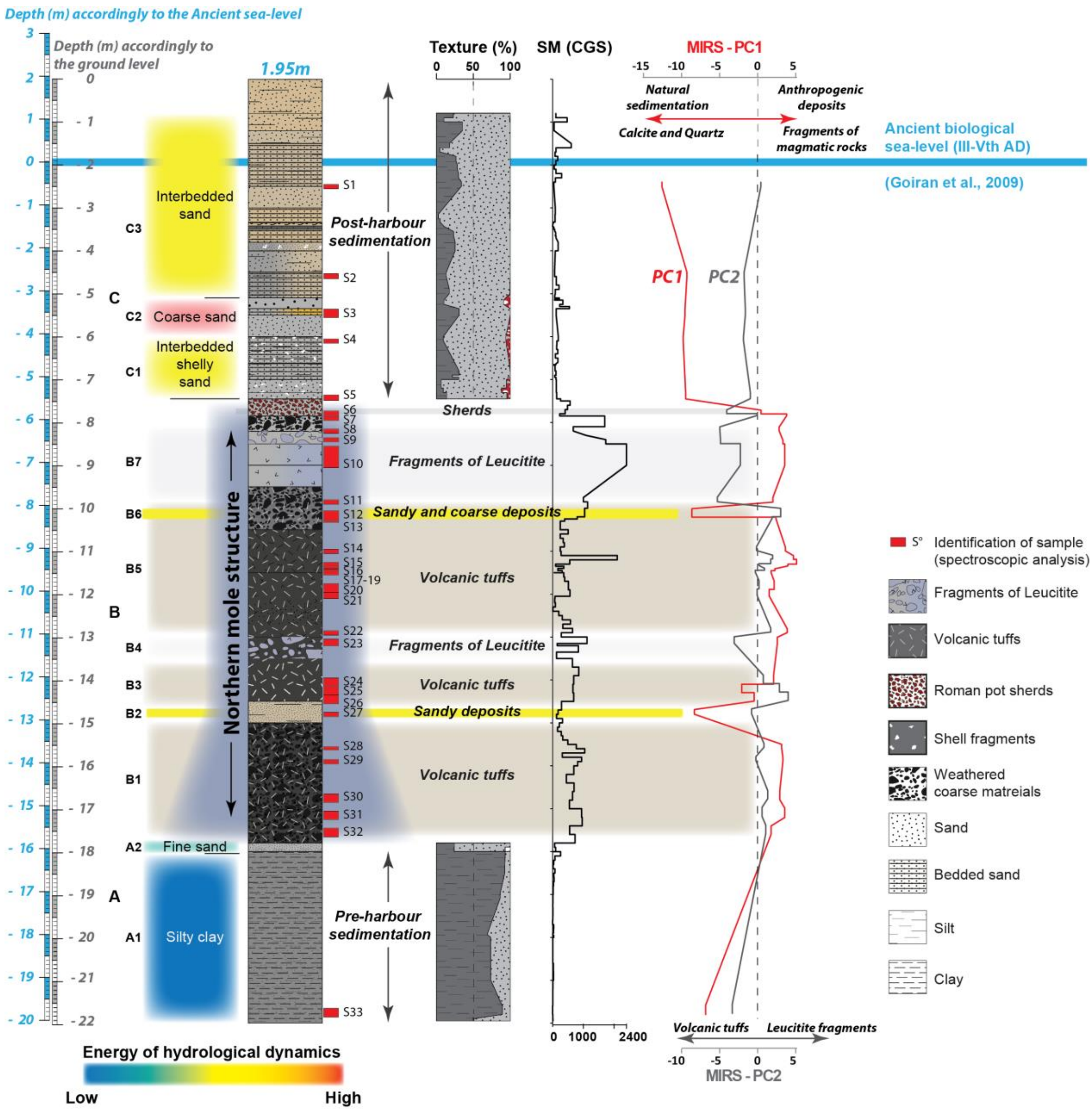

Figure 3: Stratigraphy of the drill core CL30. The figure displays: (i) texture of sediments, (ii) description of raw material used for the harbour mole, (iii) classification of the stratigraphic units and their respective hydrodynamics, (iv) magnetic susceptibility and (v) variations of the first and second axis of the Principal Component Analysis scores obtained by the infrared spectra.

3.2. Peak identification of mid-infrared spectra

Figure 4 shows the absorbance spectra of the: (i) raw construction materials and natural sedimentary deposits from borehole CL30, (ii) local outcrop references and (iii) pure minerals aiming to identify the crystal structure leading to absorbance peaks of the studied samples. Specific minerals (sanidine, pyroxene, biotite and augite) were selected and integrated into the database following previous mineralogical investigations of tuffs used in Ancient Roman constructions (Panei, 2010). Spectra of natural samples contain wide absorbance wavelengths corresponding to carbonates from 1550 to 1300 cm^{-1} (maximum of absorbance at 1430 cm^{-1}) and quartz at 1060 cm^{-1}, 776 cm^{-1}, 696 cm^{-1} and 453 cm^{-1}. Spectra of raw materials show wide absorbance wavelength range in the region from 1060 cm^{-1} to 914 cm^{-1} with specific peaks at 1012 cm^{-1}, 993 cm^{-1} and 987 cm^{-1}. Significant likeness between the raw material from borehole CL30 and the local outcrop references is related to the absorbance peaks at 1012 cm^{-1}, 993 cm^{-1}, 987 cm^{-1}, 962 cm^{-1}, 906 cm^{-1}, 876 cm^{-1}, 848 cm^{-1}, 711 cm^{-1}, 692 cm^{-1}, 470 cm^{-1} and 420 cm^{-1}. These peaks reveal spectral similarities between the raw construction materials and the volcanic tuffs in the vicinity of Rome. Moreover, comparison of these peaks with the mineralogical spectroscopy database indicates the presence of phyllosilicates (biotite-mica), inosilicates (pyroxene, diopside), tectosilicates (quartz and plagioclase feldspars), nesosilicates (sillimanite, olivine, garnet) and phosphates (apatite). To improve the recognition of spectra specific variations, a principal component analysis (PCA) was performed on the continuous MIR range (4000 – 380 cm $^{-1}$). This revealed the natural clustering of samples.

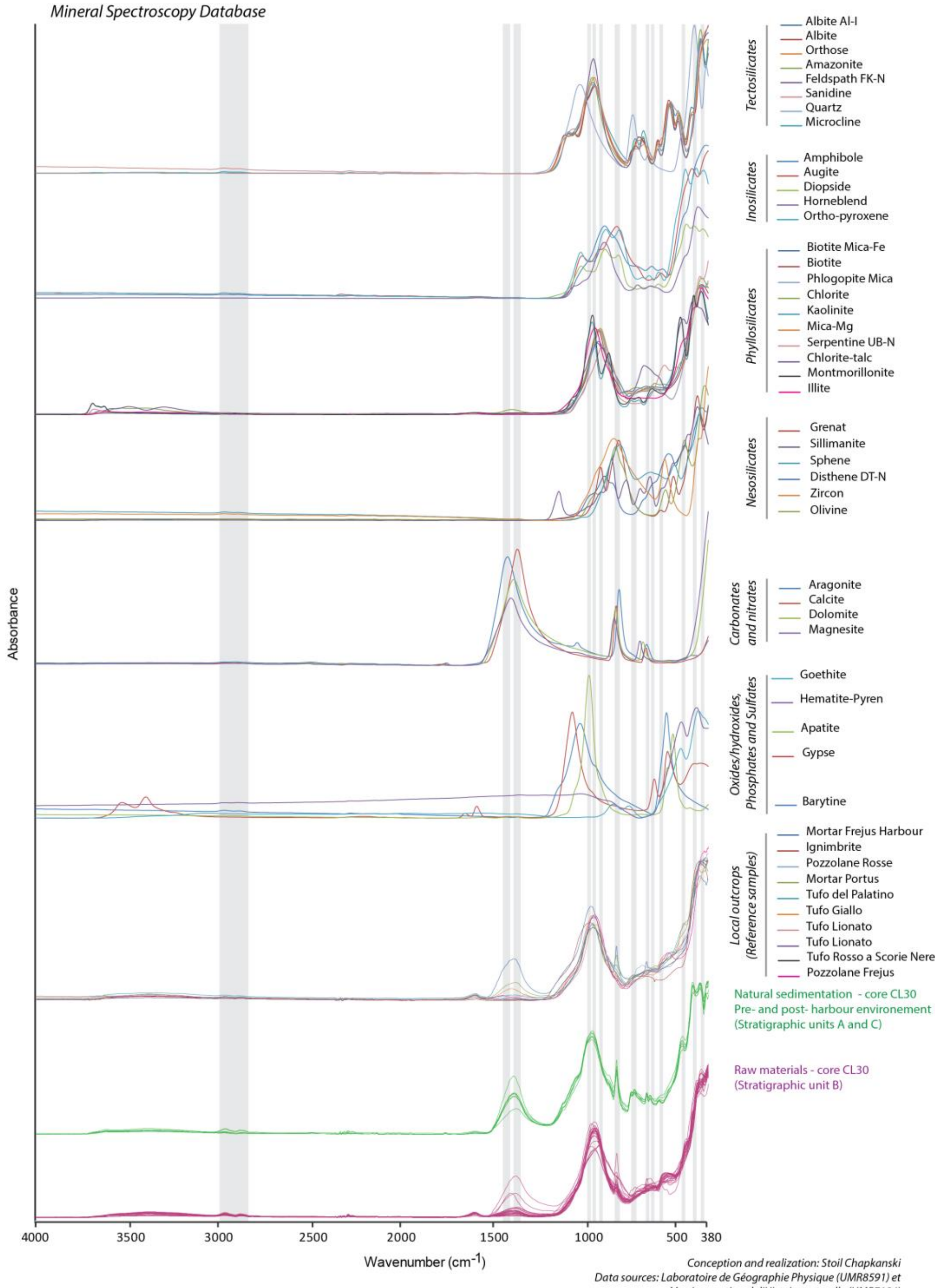

Figure 4: Overview of the mineral spectroscopy database showing: spectra of pure mineral classified by groups in comparison with the spectra from the current study including local reference outcrops, natural pre- and post-harbour sediments as well as raw construction material both obtained from core CL30. Grey lines are provided to improve the identification of absorbance peaks and to evidence spectra resemblance between the samples.

3.3. Clustering and natural pattern of spectra

The natural clustering of samples was evaluated using the principal component analysis (PCA, Figure 5). The PCA have produced seven axes explaining 100% of the total variance (PC1 61%, PC2 13%, PC3 9%, PC4 6%, PC5 3%, PC6 2% and PC7 2%). Figure 6 presents a two-dimensional scatter plot of scores for the first two principal components PC1 and PC2. These latter summarize 74% of the explained variance and reveal significant clustering between the samples. According to the first axis (PC1), samples from the natural deposits (Units A and C) form a group that opposed to the samples from the mole structure (Unit B). Moreover, the second axis reveals two sub-clusters pointing spectral similarities with samples "*Tufo Lionato*" from one side and samples of "*Tufo del Palatino*" as well as "*Tufo Rosso a Scorie Nere*" from another side. The PCA plot reveals some outliers as the samples from the Roman Harbour of Frejus (Mortar and Pozzolana), "*Tufo Giallo*" and Mortar from the harbour of Portus, that show no specific similarities with the investigated raw construction materials.

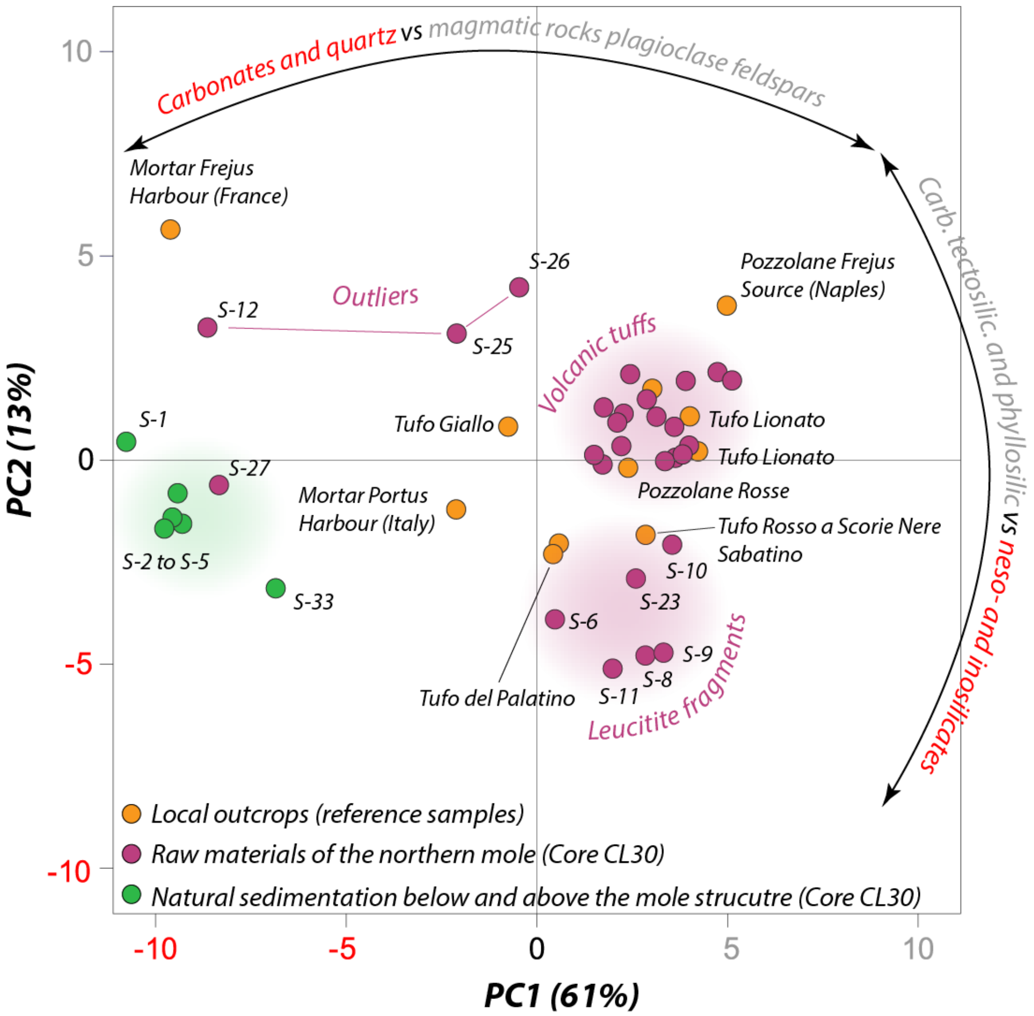

Figure 5 : Two-dimensional scatter plot of the first and second axes derived from the Principal Component Analysis of the CL30 samples and the local reference dataset. Mineral and/or association of minerals having importance of the sample clustering are indicated along the black arrows themselves placed horizontally to the respective axis PC1 and PC2. Identification of samples are provided when it was possible in order to to track their stratigraphic position (Figure 3), their spectra (Figure 4) and their visual observations.

4. DISCUSSION

4.1. *Spectroscopic fingerprint of raw materials and sediments from core CL30*

The present study demonstrates the relevance of mid-infrared spectroscopy to characterize the raw materials used in the construction of the northern mole of Portus, as well as the natural sediment deposits occurring below and above the harbour structure (pre - and post - harbour environment Goiran and Morhange, 2001, Goiran et al., 2015). Each individual spectrum contains information about the mineralogical assemblage of samples (Farmer, 1974). Following the infrared spectral fingerprint (Figure 4), deltaic sediment deposits that preceded and succeeded the harbour construction (Figure 3) consist of complex mineralogical assemblage including carbonates (calcite and aragonite), tectosilicates (quartz, feldspar), nesosilicates (garnet and olivine) and inosilicates (orthopyroxene). These latter reflect the mineralogical fingerprint of the Tiber River watershed (Bellotti et al., 2007; Bozzano et al., 2000; Tentori et al., 2018). The presence of carbonates, especially aragonite and calcite could be associated to the shelly rich sediments (Kennedy et al., 1969). The spectra of raw construction materials bear less spectra various. The mineralogical assemblages consist of inosilicates (pyroxene, diopside) and tectosilicates (quartz, albite, microcline and sanidine, also observed by Panei, 2010, Jackson et al., 2009). Carbonate contents, especially calcite, are low excepting 2-3 samples in the middle of the mole structure (around -14 m and -15 m below the ground level) consisting of sand deposits in-between volcanic tuffs. However, although carbonate content in volcanic tuffs is low, their presence could be explained by the location of the Alban Hills magma chamber in carbonate rich crustal rock (Marra, 2001). Following the spectroscopic fingerprint of the local volcanic tuffs, only the spectra of *Tufo Giallo* and to a lesser extent of *Tufo del Palatino,* indicate noticeable carbonate absorbance. Although the spectra interpretation of absorbance peaks provides valuable information about the mineralogical content of samples, it is often difficult to consider the multitude of inter- and –intra group variations induced by the absorbance peak of spectra. Therefore, this study provided a PCA in order to better understand the natural clustering and mineralogical similarities of spectra (Pezzolo et al., 2018).

4.2. *Stratigraphic interpretation and provenance insights*

At the base of the core CL30 sequence drilled into the buried northern mole of Portus, the massive grey silty-clay (Unit 1) presents natural low-energy marine environment in pro-deltaic context occurring at the time of the Roman harbour construction (pre-harbour sedimentation). The PCA scores of the spectrum from this unit indicates considerable terrigenous sediment flux and therefore consolidate the interpretation of proximal pro-delta environment. Unit A is overlaid by crumbly weathered volcanic tuffs (Unit B1) probably corresponding to low quality materials used at the first stage of the mole construction (Figure 6, samples S – 30 and S – 32). Moreover, the magnetic susceptibility as well as the PCA scores of spectra shows few variations in-between the samples suggesting the usage of homogenous materials from a mineralogical point of view. Following the PCA two-dimensional plot (Figure 5), these tuffs have a close mineralogical resemblance with the local tuff reference samples, especially *"Tufo Lionato"* and *"Pozzolane rosse"*, and are characterized by higher proportion of plagioclase feldspars. Unit B2 consists of a yellowish silty sand of low magnetic values and high calcite and quartz content. This sand occurs naturally in floodplain contexts that is also suggested by the PCA scores regrouping S-27 with the other natural sediments (Figure 5). Unit B3 consists of hard orange-yellowish tuffs with relatively high calcite content. They have been classified as outliers because of a lower mineralogical resemblance with the other materials. These tuffs are overlaid by fragments of magmatic rock that have been identified as leucitite. Following the spectroscopic data, the fragments were mainly differentiating by higher content of neso- and inosilicates comparing to the volcanic tuffs that have a high content of tecto- and phyllosilicates as well as low carbonates contents. Unit B5 is formed by relatively homogenous materials except the highly magnetic S-14 sample. The spectra have a close resemblance with the *"Tufo Lionato"*. Unit B6 consists of sandy and coarse weathered materials that have a mineralogical resemblance with the natural sediments rather than with the volcanic tuffs. These materials precede unit B7 consisting of massive leucitite blocs widely exceeding the drill core diameter. It could be supposed that the top of the mole was consolidated with larger and harder blocks. These were then overlaid by coarse unidentified materials having however a close mineralogical resemblance with *"Tufo Lionato"* and *"Pozzolane rosse"*. Romans pottery sherds occur at the top of the harbour structure.

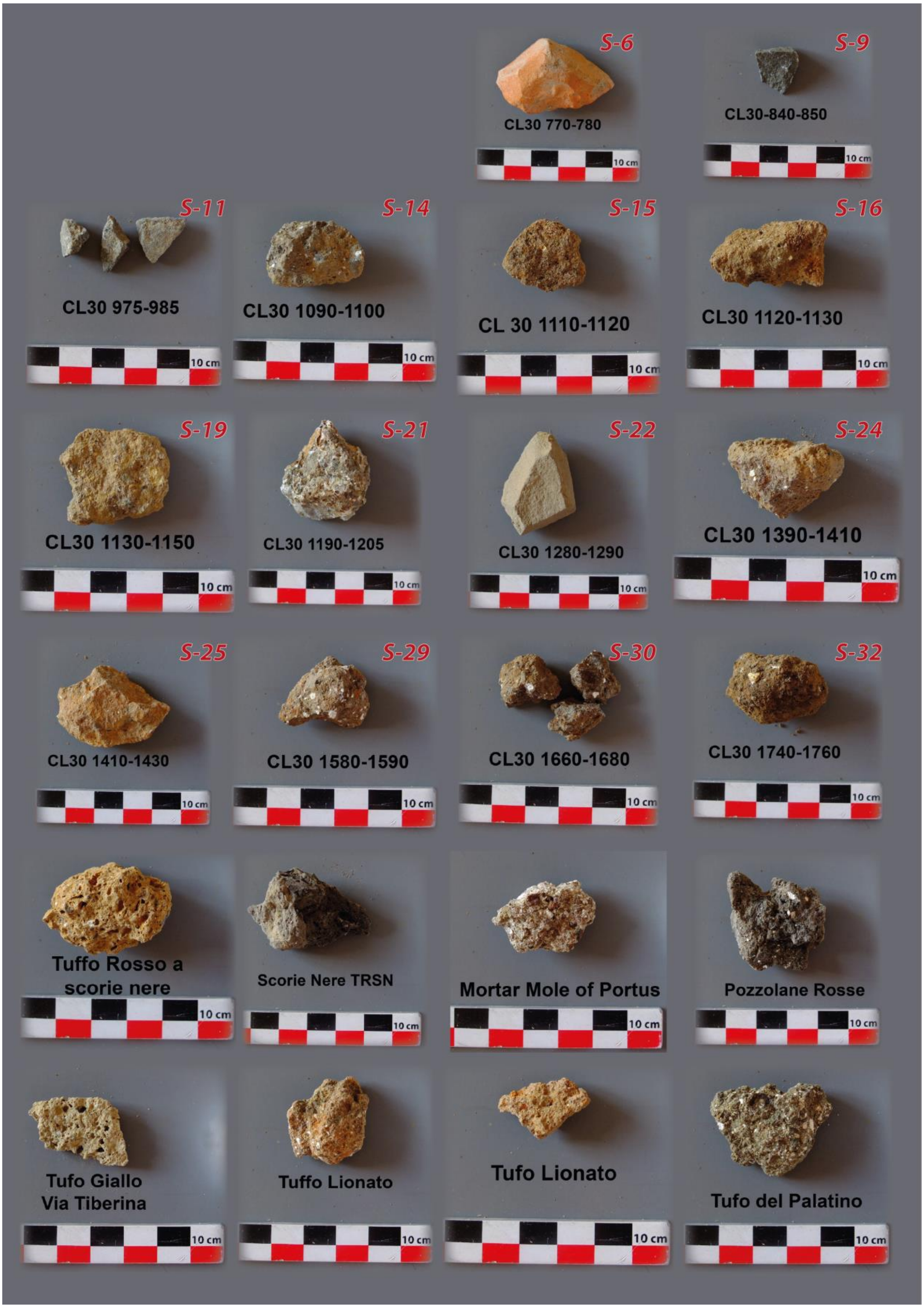

Figure 6 : Plate of photos providing visual examples of the raw construction material in comparison with the local outcrop reference samples. Sample identification and position along the stratigraphy column are displayed. The depth is expressed in cm below to the ground level.

4.3. *Methodological and interpretative limits*

The present study allows a better understanding the composition and provenance of raw materials used by the Romans to construct the northern mole of Portus, however several questions and methodological limits have arisen in the research.

The use of a mechanical drill core with an outer core liner allows deep sediment and rock investigations where archaeological excavations are limited by the phreatic water level and the depth of the trenches (Goiran et al., 2010). However, drill cores are 10 cm wide and often provide a limited understanding of the sub-surface sediment archives. Therefore, drill cores should be multiplied in order to better understand the shape of the structure and its spatial extension. The stratigraphic interpretation of the core CL30 provides an insight to the raw materials that have been used in the construction of the northern mole. Moreover, the usage of crumbly and low quality materials at the basis and hard and solid builders at the top, suggests a specific building strategy. However, it is impossible to extrapolate these finding and consider that the mole has been built with the same materials and in the same way. Also, some boulders widely exceed the diameter of the drill core making impossible to estimate the size of raw materials. Therefore, research studies involving more drill cores and archaeological excavations are in process and focus on both northern and southern moles of Portus.

Another fundamental question is related to the lithological sources of the raw construction materials. Although the present study demonstrates clear mineralogical resemblance between the materials from core CL30 and the outcrops of *"Tufo Lionato"* and "*Pozzolane rosse*", sampled near Rome, this does not allow to establish a simple connection between the primary sources and the last deposits. It is possible that the construction materials have been reused from former Roman buildings. Despite these limits, the mid-infrared spectroscopy coupled with PCA prove to be a relevant tool to investigate the provenance of raw construction materials.

5. CONCLUSION

This research study aims to provide insights about the lithological provenance of the raw construction materials employed during the construction of the northern mole of Portus, the Imperial maritime harbour of Rome. To do this, a multidisciplinary approach involving deep mechanic drilling into the mole structure, sedimentological analyses and mid-infrared spectroscopic measurements were performed. Infrared spectra were obtained from pre- and post-harbour sediments as well as from mole's raw construction materials and were compared to both mineralogical spectroscopy dataset and spectra from local tuff and Pozzolana using multivariate statistics. Although the comparison of construction material and natural outcrops involves several limits, discussed above, the results and interpretations were considered with care and permitted to: (i) characterize the mineralogical content of sediments and raw construction materials of core CL30, drilled into the buried structure of the northern mole of Portus and (ii) provide insights about their lithological provenance at the basis of reference samples from the vicinity of Rome. The results of the study revealed close mineralogical resemblance between the majority of raw construction materials and the *"Tufo Lionato"* and in a lesser extent to the "*Pozzolane rosse*". Moreover, leucitite fragments have been identified and emplyed principally in the top of the harbour structure. This study constitutes a starting point for further provenance investigations of materials used during the construction of the Portus harbour.

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