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# Recording invisible proofs to compose stone narratives.

# Applications of Near Infrared Spectroscopy in provenance studies.

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## Abstract

The history of human-environment interaction is embedded in stone. Stones are essential components of daily life and their various usage characterize certain areas or chronological periods. The form of a stone object is the result of a long chain of interactions with distinct bodies but the intangible life story of any artefact is partially registered in its original material properties and gradual physical alteration. Digital systems can be adopted for collecting these invisible records and tracing a stone's history. Chemical imaging and portable spectroscopy are quick and non-destructive remote sensing techniques that can be used to gather empirical data and track production and use of stone artefacts over time. This article reviews the application of Near Infrared Spectroscopy as a method for geochemical characterization of objects and as a tool for provenance studies within the Mobima project; carried out by an interdisciplinary team of archaeologists and chemists at University of Umeå, Sweden.

Near Infrared Spectroscopy can be used for acquiring and processing spectral information directly in the field, modelling datasets of big assemblages and classifying objects. Making stones' biographies visible will help understanding the entanglement of past societies and their geological landscapes.

## Introduction

The life story of a stone artefact is an assemblage of events evident in the tangible and intangible attributes of the object. By the term tangible, I generally define attributes that are visible to naked eyes like the color or the shape; other qualities, such as the cultural meaning that the artefact assumed in a certain context, are invisible, and therefore intangible; finally, some attributes related to the geochemical characterization of the material are invisible but can be recorded through analytical methods. Investigating the geochemical characteristics of stone artefacts is essential in order to read the complex network of agents co-occurring in an object's biography (Kopitoff, 1984). Digital tools can be beneficial in order to decipher some of these evidences. Technology helps with reconstructing narratives from otherwise invisible proofs, in particular,

the application of portable Near Infrared Spectrometry and Imaging represented a turning point in the archaeological practice. This technology, particularly useful to gather large geochemical datasets, has been successfully applied to the study of various archaeological features at different scales, from landscapes survey to characterization of lithic tools.

In this paper I discuss the epistemological implications with the study of stone artefacts referring to new materialistic positions and the broader debate over theoretical issues within archaeological sciences (Boivin 2005, Jones 2004, Hodder 2012, Pétursdóttir 2012, Ruggles and Silverman 2009, Tilley 2004), related to that I describe different case studies in which the use of technology supported the experimentation of new protocols for acquisition and processing of empirical data. From in-field measurements of soils and pigments, to hyperspectral imaging of ancient walls and pedagogical implications in teaching archaeometry, I will consider potentialities and concerns with the use of spectral remote sensing. All the case studies presented are carried out within the Mobima project<sup>1</sup> (Mobile Imaging in Archaeology), which goal is testing and developing applications of short-range remote sensing techniques on archaeological materials with a specific focus on Near Infrared Spectroscopy (NIR).

## Theories for disentangling an "ecology of things" (Bennett 2009)

The use of stone materials for various purposes is a constant throughout the entire history of humanity. The mineral world has been quotidian to humans since prehistory as a long-term entanglement: from hunter-gatherers' knapped tools to more recent mines, quarries, and buildings [Foley and Lahr 2003] [Waelkens et al. 1992]. From early humans up to this day, stone materials come in different forms, textures, sizes, and correspond to precise needs. The selection of particular raw materials supply sources is mostly determined by an empirical observation of rock's chemical and mechanical properties. For example, a certain type of marble has been suitable for a particular sort of art and architecture, obsidian appears to be an appropriate material for blades and spear tips, while pozzolana sand is good for hydraulic mortars. Other contingent factors equally occur in the choice such as the range of raw materials naturally available, the network of trades/exchanges and the accessibility of the bedrock. Considering these circumstances we can affirm that a stone artefact is always the product of the joint agencies of human and stone mutually influencing one another.

As Tilley states "social relations are simultaneously relations between material forms" [Tilley 2004, 217], as a consequence of that, the study of socio-cultural dimensions of stone artefacts should also be based on the analysis of their structure and physical properties. Material and physical agents together with social practices are co-occurring in the network of human-stone interaction [Pétursdóttir 2012]. A stone artefact encloses the "objective nature of material culture and the subjective experience of human beings" [Ruggles and Silverman 2009, 11], as the product of a long chain of interactions with other bodies generating variations in their material tangible properties: rain, frost, and sun provoke geochemical modifications on stones leading to decay and erosion; a Roman marble inscription celebrating a victorious emperor can be cut and

<sup>&</sup>lt;sup>1</sup> http://www.idesam.umu.se/english/research/research-projects/mobima--mobile-imaging-in-archaeology/

reused as building material for a Romanesque cathedral. Various agencies are entangled and can be categorized by using different chronological and spatial scales. For example, usage of raw stone materials can be mapped by reconstructing the steps of exploitation and colonization of natural resources in a region, while the network of trades and transport is taken into account by tracing movements of people and stone goods over longer distances. Mechanical properties of different stone types are optimized in order to get stable and resistant artefacts in accordance with their available and contemporary technology. Finally, stone artefacts can carry a cultural meaning deeply connected to their physical being as particular objects in ritual contexts.

The combination of a stone's tangible and intangible properties is better understood via its materiality, defined as: "*a notion that encompasses the view that material and physical components of the environmental and the social practices enacted in that environment are mutually reinforcing*" (Jones 2004, 330). Every object is part of a texture of matters, as outlined by Hodder (2012), and the interactions between humans-artefacts-environments can be read from the point of view of things. The focus on an object's materiality shifts the archaeological perspective on cultural interpretation; the artefact is no longer a clue to be decoded but an essential component of the past characterized by material properties that must be explored in detail (Boivin 2005, Jones 2004). To which extent can the study of stone artefacts' materiality depend on the analysis of their physical properties? The socio–cultural environment and the geological characteristics are equally necessary to describe an artefact. In this sense, cultural interpretation and archaeometric data are necessarily entangled.

Various terms have been used to refer to the study of geochemical characteristics of stone artefacts. These terms often carry implicit theoretical connotations. The debate over methods for collecting data concerning the geochemical characteristics of objects is developed in the field named provenance studies and generically defined by Malainey (2011) as the study of attributes suitable for tracing the raw material the artefact was made of and where it was manufactured. Although the words provenience or provenance are equally adopted to define this particular branch of archaeology, the use of one or another term is ambiguous. A debate has been undertaken by scholars suggesting that the terms provenance and provenience actually refer to different concept and not only a spelling variation<sup>2</sup> (Boivin 2005, Joyce 2012, Jones 2004, Malainey 2011, Price and Burton 2011). The distinction between provenience and provenance is often related to the context. For example, when associated with museum and archive policy for item acquisition, the word provenance implies "the history, the life of the object" including previous owners, while provenience is the "find spot" (De Angelis 2006, 398). In relation to archaeological research, provenience is defined by Price and Burton as either "the place of discovery" of an object or its "place of origin" (Price and Burton 2011, 213). I refer at provenance and provenience as indicating two different methodological and theoretical strands.

In the US, the term provenience is used by archaeologists and chemists principally indicating the place of origin of the raw material an artefact is made from. A consequence of this concept is the provenience postulate that states that: "*the chemical differences within a single source of* 

<sup>&</sup>lt;sup>2</sup> According to the Oxford dictionary the two terms are synonymous and there is not a relevant difference in the meaning http://www.oxforddictionaries.com

*material must be less than the differences between two or more sources of that material, if they are to be distinguished*" (Price and Burton 2011, 214). In these terms, the study of an artefact's provenience poses geochemical analytical problems, especially for the definition of the material's signature. Consequently, the success in the application of the postulate is uncertain from case to case and it depends on: type of materials, geographic distribution of sources, analytical techniques, budget/costs, time, and type of artefacts (Price and Burton 2012, Malainey 2012). Nevertheless, a strict focus on the analytical issues related to the application of the provenience postulate risks resulting in a never-ending debate on different methods' technical details. The use of analytical methods must be related to the transformation of the paleo environmental record and the problematics of artefacts' proveniences need to be contextualized in the site development dynamics.

I apply the term provenience to indicate the field of studies that aims at finding the origins, the sources of the raw materials, marking a direct thread which ties together supply-artefact and relies mostly on geochemical analytical techniques. Provenience is a markedly static concept, which does not include the interaction of the object itself and the environment. Nevertheless, the origin of raw materials is to be considered part of a more comprehensive narrative of provenance: the chain of events that shaped an artefact the way it looks like when we observe it. Objects are constituted primarily of raw materials and are continuously altered changing their properties and becoming other things (Joyce 2012, 124). The ecosystems, in which artefacts and sources are situated, play an important role in defining a stone's geochemical signature as they are in continuous change. Quarries, mines or outcrops are exposed to weathering and buried sites are influenced by post depositional processes (modification occurring to the sediments once they are in place), both of which are capable of causing chemical alteration of the geological materials. Environmental and human factors can make objects moving. Erosion, water transportation or human activity can carry artefacts far from their place of production. All these movements and transformations might interfere with a clear identification of the geochemical characteristics of objects and sources, but are fundamental for defining the biography of the artefacts.

Provenance is therefore more appropriate to refer to the life of the items in context, it applies to the entangled history of humans and stone materials as traced by the co-occurrence of physical characteristics and acquired meanings. Digital tools can help making visible the proofs of ongoing narratives, expressed in objects' intangible properties.

#### Methods for assessing the invisible

Artefacts are not crystallized traces of human activities but active players in dynamic environments, referring to the interaction of distinct objects on different scales of matters (Bennett 2009). The concept is not new in archaeology and it can be backtracked to Schiffer's work (1987) on the *Formation processes of the archaeological record*. Although Schiffer makes a distinction between systemic contexts and archaeological contexts, somehow a limitation to the hypothetic interactions between humans and things, he classifies the processes that influence artefacts in a physical way. Variations in the material attributes of objects should be recorded and linked with the biography of the artefact itself, defined as provenance. Measuring artefacts' properties is a fundamental step for understanding the stories of their provenances. This process of mapping interactions is extremely challenging due to the high variability of physical modification that may occur to things and that things contribute to. Considering the material qualities of artefacts as the center of a network of social relations, symbolization, physical interactions with the environment and subsistence, we are able to formulate more interesting research questions within a more comprehensive theoretical frame (Dunn and Woolford 2012; Dunn *et al.* 2012). A solid narrative of provenance relies on the association of epistemological issues, sampling strategies and tools for the collection of empirical data. A theory of things based on objects' physical properties is linked to the discussion on methods for gathering and processing information.

Digital techniques, analytical tools and sampling strategies applied in different case studies then must be calibrated on the target, adapted to record the complexity of the network between humans-things and environment. The physical and material properties of a stone artefact are measurable attributes, and observations that can be conducted refer to standard systems: the geological description (petrographic-mineralogical), the chemical characterization (elements and properties, carried out using various methods) and the documentation of traces resulting from the contact with other bodies (tool marks for example). Consequently, analytical tools can be designed to fulfill the demand of a certain type of empirical data (Jones 2004; Bray and Pollard 2005), avoiding the risk of generating datasets not comprehensive of the set of issues that are the focus of theoretical practice (Boivin 2005).

A substantial problem for the determination of stone materials' characteristics with geochemical quantitative analysis is represented by the choice of sampling strategies. Geochemical methods applied in provenance studies (petrographic analysis, X ray fluorescence, mass spectrometry, instrumental neutron activation, just to mention some of them) are often destructive, expensive and time consuming. Analytical techniques carried out in laboratories require a careful selection of the objects to be studied. The system applied is often a convenient sampling method based on project specific needs and related research questions (Orton 2000). I argue that this could result in a biased dataset representing some specific targeted queries to the detriment of the evaluation of the entire variability of the objects' population. In selecting variables to record and items to measure, archaeologists are influencing the questions that can be posed to materials (Boivin 2005), testing tools for recording and visualizing the material properties of a more comprehensive range of artefacts is essential in order to defy workflows based only on destructive and punctual analysis.

The choice of using Near Infrared Spectroscopy relies on practical and analytical strategies. NIR is a technique based on recording different molecular vibrations in the Near Infrared band of the

electromagnetic spectrum. It is a non-destructive method that requires the use of controlled light source and digital captors (like cameras or probes) and that can be implemented in various contexts. NIR covers the spectrum region between 700 and 2500 nm and contains absorption bands corresponding to overtones and combinations of fundamental C-H, O-H, and N-H vibrations. NIR has a scarce sensitivity to minor constituents, as a Near Infrared Spectrum doesn't present clear peaks but often it shows more broad bands that are the result of aligned peaks (Bokobza 1998). Considering NIR properties, the identification of elements from a single spectrum is often not worthwhile and a fingerprint approach is preferable. One big advantage with the use of NIR is the fact that it is a nondestructive method requiring a very simple sample preparation, and performs long series of measurements in short time. This makes it a method with interesting possibilities of application in archaeological research, recording spectra from various elements several times. NIR is largely used as a control test in pharmaceutics industries, agriculture, food production and geology. For example, NIR has been applied to a wide range of geologic problems: mineralogical mapping of lunar surface (McCord and Adams 1973), mineral exploration (Goetz et al. 1983), mineralogical analysis of drill cores (Kruse 1996), field mapping of expansive soils (Chabrillat et al. 2002) and field mapping of mineral assemblages for gold deposits explorations (Bierwirth et al. 2002). The development of close range hyperspectral imaging associated to LIDAR scanning has allowed rock characterization of quarry walls both with natural and artificial light. Mineralogical variation in vertically oriented rock faces has been mapped at high resolution using a light weight hyperspectral imager (Kurz et al. 2012). Moreover, several spectral reference libraries are already available as open source from various agencies. The USGS has a database consisting of over 1300 spectra available<sup>3</sup>, the library contains minerals, man-made materials, vegetation and other materials. Jet Propulsion Laboratory ASTER Spectral library version 2.0<sup>4</sup> contains more than 2400 spectra of minerals, vegetation and man-made materials.

Mobima project's target is to construct an experimental analytical protocol for archaeological materials which could be applied to features directly in the field or to large datasets of artefacts in laboratory. There are various portable instruments on the market which allow us to multiply the number of measurements and bring the laboratory directly to the field (for example portable X ray fluorescence or portable Raman spectrometers), due to the constant development of tools for non-destructive analysis and handheld devices. On the other hand, as a consequence of the use and misuse of these methods, an opinion has spread that portable instruments are not sufficiently reliable for chemical elemental characterization of archaeological objects (Fraham 2014, Shackley 2012, Speakman and Shackley 2013). This opinion of portable instruments is likely due to an insufficient evaluation of the data gathered and the instrument settings/calibration, which had as outcome the production of datasets unacceptable in terms of accuracy for fulfilling the provenience postulate.

<sup>&</sup>lt;sup>3</sup> http://speclab.cr.usgs.gov/spectral-lib.html

<sup>&</sup>lt;sup>4</sup> http://speclib.jpl.nasa.gov

By using field-portable and quick image-based techniques, it is possible to get a more global overview on materials' surface chemical characteristics. NIR can be used as a unique tool for categorizing different types of raw materials (using spectral data as common fingerprints for same source artefacts) or as an exploratory method to set sampling strategies for elemental analysis. The adaptability of NIR instruments makes it possible to be applied to almost every kind of material. The extended collection and interpretation of spectral data could become a digital added value to an artefact's optic description.

Within the Mobima project two different devices are used for testing Near Infrared Spectroscopy on archaeological materials.

- 1. The portable micro probe produced by Jdsu (now Viavi) covers a wavelength range of 908-1676 nm. The probe has two integrated tungsten lamps and a captor that is 5x3mm wide. The probe is connected to a tablet used to manage and store the records. The instrument is calibrated every 5 minutes (on average) using a white and a black reference (see figure 1, on the left). The portable probe is suitable for research directly in the field, is easy to carry and can record spectra in few seconds.
- 2. The Sisuchema Pushbroom short wave infrared hyperspectral imaging system (1000–2498nm) is a non-portable Near Infrared (NIR) imaging systems used in the laboratory mounted on a conveyor belt for taking pictures of large artefact datasets or sample series (see figure 1, on the right).



Figure 1\_ The Viavi micro probe used for recording spectral data on structures discovered during survey in Auvergne, France (on the left). The Sisuchema Pushbroom hyperspectral camera in the laboratory at Umeå University while scanning Mesolithic quartz tools (on the right).

A parallel experiment is carried out testing the use of a modified reflex camera for field imaging. By removing the blocking filters and adding two different NIR filters (720 and 950 nm), it is possible to transform a digital SLR camera into a tool for NIR multivariate imaging. The spectral information obtained with this instrument is not as detailed as that collected with the Hyperspectral imager, but it can still be used as a quick data evaluation technique (details in Geladi and Linderholm 2015, Allios et al. 2016). These instruments in combination grant a good coverage for collecting Near Infrared spectra both in the field and in the laboratory. Both the NIR probe and the Hyperspectral camera work connected to a tablet or a pc so that the data can be evaluated directly during the field/lab operation.

NIR potentialities and limits for archaeological research can be evaluated referring to the two instruments tested within the Mobima project (JDSU microNIR and Sisuchema pushbroom, mentioned above) according to precision, accuracy, reliability and validity<sup>5</sup>.

Working with the Micro NIR in the field, there might be a precision problem with the positioning of the probe on the surface of the materials. The captor window inside the instrument is usually small compared to the holder (4x2 mm, placed in the center of a 4 cm wide holder): for this reason addressing a measurement on a very specific spot becomes problematic (considering also the non-homogeneity of many rock types). The calibration in the field is performed using a white reference (100% reflectance) made by spectralon, a fluopolymer with the highest diffuse reflectance of any known material in the near infrared region of the spectrum. For the black reference (0% reflectance), the producers' recommendation for the JDSU probe is to calibrate by pointing the spectrometer in a dark corner. In field conditions finding a spot with no natural or artificial light disturbance might be difficult, therefore an easy solution has been adopted placing the probe on a sheet of fine grained black sand paper.

Accuracy of NIR portable spectrometers and imaging systems can be evaluated within an internal validation model and in comparison with other non-destructive and portable techniques. The method that has shown to be suitable for the correlation, accordingly to our project aims and goals, is portable X-Ray Fluorescence spectroscopy (XRF)<sup>6</sup>.

<sup>&</sup>lt;sup>5</sup> These concepts have been codified in the 19<sup>th</sup> century by measurement and evaluation theorists and applied for obsidian provenience studies (Fraham 2011; Huges 1998). Here a quick glossary:

**Precision**: We consider precision as the relative standard deviation for repeated measurements on a sample (Fraham 2011). The measure of systematic error and random error can be considered as a variable when calculating the reproducibility of a set of measurements.

**Accuracy**: Accuracy of a method is to be checked against specimens with known composition from a recognized organization. Furthermore, samples must be analyzed using two or more analytical techniques.

**Reliability**: Reliability involves precision and accuracy (Huges 1998) and it refers to the difference in measurements in different conditions over time. In these terms, reliability can be quantified on a population as relative standard deviation or as a test for statistical difference (Fraham 2011).

**Validity:** The definition of validity in literature corresponds to two different levels. There is a quantifiable validity coinciding to accuracy and defined as a constant standard deviation (systematic error) that can be corrected, and a notional one referring to the ability of an instrument to record values that are relevant for the study target (Frahm 2011). According to Carmines and Zeller, it is not possible to validate the instrument itself but that must be done in relation to the specific purpose for which it is applied (Carmines and Zeller 1979).

<sup>&</sup>lt;sup>6</sup> Another interesting correlation could be done associating NIR and Raman spectroscopy for the study of stone materials in the field. Raman is a vibrational non-destructive spectroscopic technique. Like NIR, this method is fast and does not require complicated sample preparation. Raman spectroscopy has been used in geology for sourcing rock materials, as the minerals can be easily identified without major problems (Jehlicka 2011). It is possible to find

XRF is a well-established technique used for sourcing materials in archaeology. Scholars have been debating the applicability of portable XRF devices for provenience studies (Fraham 2014, Shackley 2012, Speakman and Shackley 2013) and the discussion about the reliability and the treatment of such data is still open. A major problem is presented by the application of calibration systems. Handheld portable XRF devices are often sold with a series of calibration models loaded in the system. The models are calibrated through a fundamental parameter routine against samples of known composition. This type of calibration is good for a presence/absence assessment but not accurate enough for provenience elemental analysis (Shackley 2012).

## NIR in practice

Within Mobima, the research focus is on method development and experimental design. For this reason, the project is run on various materials and sites. Portable NIR spectroscopy has proved effective in relation to some case studies already published and in particular for stone materials characterization (Allios et al. 2016, Linderholm et al. 2013, Linderholm et al. 2015).

The first application of NIR in archaeology within this framework was the study run on bone fragments classification in archeological soils by means of NIR. The NIR hyperspectral imaging was applied to the identification of animal bone material in complex sieved soil-sediment matrices from a mid-Mesolithic site in northern Scandinavia. The whole dataset of bones and teeth samples was analyzed through image based nondestructive techniques and used as reference for the identification of fragmented skeletal material on soils from the excavation. The information about the bones' chemistry-mineralogy variability within the site and in different layers provided information about the state of preservation of the materials (Linderholm et al. 2013).

A field based study of rock paintings in northern Scandinavia was carried out during the autumn of 2014 using the NIR portable Micro probe. The detailed results from the targeted analysis on a panel in Flatruet, Härjedalen, Sweden show a large spread in spectral data from red paintings and the background. Nevertheless, evidence of clustering was observed and using multivariate data analysis and it was possible to separate the information related to the paintings. Using partial least squared discriminant analysis and setting bedrock and paint as discriminant variables, the separation was clear and the model was used for a classification test. The classification was run on 20 samples (10 paint and 10 background) and only three spectra were misclassified. An analytical problem emerged due to the spread of secondary iron oxides from precipitation on the surface of the rock wall but this resulted in measurement noise rather than a real item classification problem. In a local model scale, looking at the different composition of pigments within two painted figures, it was possible to spot the difference between two different elks feasibly painted with different red pigments (Linderholm et al. 2015). This study opened the way for further on-site analysis of painted prehistoric panels, highlighting some analytical problems and showing how they could be overcome.

on the market miniaturized portable Raman spectrometers, but so far there are few publications evaluating the use of these devices (Edwards 2012). In contrast to NIR Raman spectroscopy requires more technical expertise but results in sharper peaks. The two methods can be considered as complementary as the excitation occurs in different vibrational states and molecular bonds that are not active within one method are generally active in the other one.

Since February 2016 a comprehensive field study has been launched on the medieval city walls of the city of Carcassonne (France). The study was initiated as an international collaboration between the Umeå University (Sweden), the French university of Rennes 2 and IRSTEA Montpellier. The goal was the application of different spectral techniques for the study of the use of raw stone materials in the fortress walls. The data were collected using a NEO HySpex-320m-e camera (1000-2500 nm), mounted on a pulley system and moving on a rail. The Hyperspectral data were associated to NIR enhanced color photography and point measurements performed with NIR microprobe and compared to portable XRF (Thermo Scientific Niton XL3t ED-XRF analyser using a 50 kV X-ray tube with an Ag target and a Si (Pin)-detector and applying the Mining Cu-Zn calibration). The dataset was processed through multivariate methods in order to evaluate the clusters and draw a first overview of the materials' provenance (Allios et al. 2016). The association of various methods allowed the understanding of the dynamics of stone use and re-use on the entire extent of the walls in different chronological periods. Such a wide perspective is crucial to understand the impact of the monumental construction in the landscape in terms of materials supply and exploitation of quarrying sites.

Near Infrared Spectroscopy has been successfully used also for educational purposes, in particular within the archaeological field school for master students in the environmental archaeology program at Umeå University. On excavation, students collect and process data in real time. This approach is pedagogic for understanding the correlation between features observed in the field and chemical properties registered in the Infrared Spectra. Non-destructive field-based analytical techniques are taught together with traditional sampling strategies and wet chemical laboratory analysis.

Applications of Near Infrared Spectroscopy in geology are mostly related to industrial and mining research, while within archaeological provenance/provenience studies elemental analysis is generally preferred. There is still some resistance amongst archaeological geochemistry specialists towards the applications of NIR for stone materials studies, motivated by the difficulties in making element identification from the spectral data. This argument is legitimate when the research goal is sourcing materials fulfilling the "provenience postulate" (mentioned above, Price and Burton 2012) only according to defined and quantified geochemical association of elements. It would be a mistake to focus on measurement precision to the detriment of the archaeological accuracy needed for a determined case study (Shackley 2005). Within the Mobima project, the Near Infrared spectra are often not evaluated for peaks identification but their shape corresponds to the combination of chemical elements constituting a single material bar code. Of course, this approach involves some inconvenience. The direct comparison of chemical elements in stone materials obtained with other analytical methods becomes arduous. Nevertheless, by multiplying the measurements and processing the dataset through layers of multivariate analysis, it is possible to create local models for clustering and sourcing.

## Data mining, data minding

NIR spectroscopy can be used extensively as a digital screening method. The application of this type of target specific portable spectroscopy or imaging techniques provides a detailed overview of the physical-chemical characteristics of the materials. NIR turns into a crucial tool for looking at the large scale variability of stone artefacts' qualities and an essential sampling tool for

elemental analysis. The application of NIR to archaeological research makes it possible to collect big datasets in short time (Allios et al. 2016, Linderholm et al. 2015). The first field test application of the NIR probe for the study of rock paintings allowed the collection of 1432 spectra in the near infrared within working five days and from six different sites (Linderholm et al. 2015). The time for each measurement is about 1-2 seconds and there is no limit to the number of records. The data collected with the Micro probe are stored as data matrix files in which the variables/wavelengths are reported. The spectra can be visualized on the laptop/tablet in real time during the acquisition.



*Figure 2\_SWIR images visualized on the laptop in real time during data acquisition (Carcassonne project)* 

The hyperspectral image files are more laborious to process. Depending on the size of the image the dimension of the data files can vary from about 50 to 500 MB (raw format). When working with the lithics from a site, for example, we have to deal with image datasets of up to 22 GB for a total of 2261 objects analyzed. For these reasons the computing time for processing information must be taken in account. The use of a large dataset means that the sampling bias becomes less influential, and the responsibility of the researchers in choosing the fragment to analyze is reduced. The procedures adopted by the Mobima Project members are based on a Big Data Approach, consisting in hierarchical and cluster models. Archaeometry, like the rest of archaeology, is involved in the *big data challenge* (Gattiglia 2015), both from a computational and a theoretical point of view, statistical and interpretative tools are improved to handle large amount of information. When analyzing a big dataset lacking in structure (meaning not

structured in a SQL database) with statistical tools we can spot similarities and patterns among the data (see figure 3). This modus operandi is usually referred to as data mining and it allows to cross reference large datasets and create clusters of aggregated data (Boyd and Crawford 2012). Within a data mining practice the information is processed in order to find correlations rather than causations (Gattiglia 2015).

In order to follow this path we apply methods issued from the data driven discipline of chemometrics, which focuses on the application of mathematical methods for analyzing chemical data. Chemometrics deals with both descriptive and predictive analysis and it is grounded especially in multivariate data analysis. Principal Component models are used as exploratory tools to spot patterns in the data matrix. Image mapping is based on cluster analysis so that similar spectral values can be projected directly on the picture. Spectral information is evaluated and associated to the spatial distribution on the object surface. The data collected are processed using the software Evince by Prediktera, which is designed for operating image mapping, spectral transformations and multivariate statistics (more details on the software at <u>www.prediktera.se</u>).

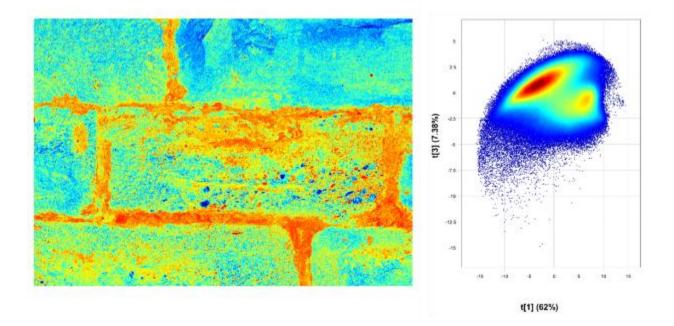


Figure 3\_Detail of the inner wall of Carcassonne acquired with the NEO HySpex-320m-e camera system. On the right the PCA model shows all the pixel values distributed in the multivariate space, on the left the same pixels in the image are colored according to the intensity value (t1). It is easy to spot the compositional difference between the building materials, in this case sandstone, and the architectural mortar. Moreover the hyperspectral image highlights some inclusion in the sandstone (blue dots in the center on the image) that can be used to establish the quarry of origin.

NIR technology associated with chemometrics becomes a powerful tool for screening and evaluating big datasets, by working on associations among different materials in a way that couldn't have been possible with other elemental analyses. The visual approach to chemical data that can be reached with these tools shifts the focus on the variability of the chemical properties of stones in a local model instead of a determination of the singular elements. The visualization of spectral data directly during the acquisition via digital tools represents a substantial difference between NIR and other analytical methods. The invisible attributes of stones can be disclosed while looking at the artefacts themselves, providing an advantageous tool for sampling and work planning.

# Conclusions

In the first section of this paper the theoretical frame of the study of stone artefacts was problematized in order to delineate a more solid protocol for tracing the provenance of objects. Moving from a theoretical body for the study of stone-human entanglement, focusing on nonhuman agencies, to the empirical data collection represents a challenge for our discipline. It was shown how the adoption of new analytical procedures could be helpful for acquiring geochemical data, in particular a sampling strategy targeted to the reduction of the influence of researchers' prejudice about materials could help focusing more on artefacts' materiality. NIR offers a range of interesting possibilities. Portable spectrometers and imaging systems are versatile instruments that can be used as short range remote sensing tools both in the laboratory and in the field. The technical peculiarity of this type of spectrometry makes it possible to distribute the measurements on the surface of the objects allowing a diffuse quantification of the chemical properties and the direct observation of their variability.

Visualizing and increasing exponentially the number of measurements performed is a critical step in order to build an analytical protocol adequate to represent the peculiarity of chemical-geological information at different scales.

The preliminary results from Mobima project show how the application of NIR technology represents a resource for looking at the intangible strands of an artefact's biography. Near Infrared Spectroscopy can be adopted as a screening technique for picturing geological materials' chemical topography, involving in the analysis information that would have otherwise been neglected. In different archaeological contexts NIR tools can provide precious information, however the application is dependent on a good understanding of the spectral signatures of various materials, together with statistical modelling.

NIR spectra datasets are useful sources of information about correlations between objects, however the archaeologists still hold responsibility for the interpretation of these patterns. Knowing what type of pigment was used in a rock painting gives us a suggestion about the painter's technological know-how and the materials she/he had at disposal, but it could indirectly contribute to the definition of a typo technological series of paintings on different sites. NIR spectra can help us understanding how much of the roman walls of Carcassonne still stand but will not suggest what happened to the rest of the blocks.

As other digital analytical tools NIR can help recording the visible and invisible traces of events embedded in the physical form of a stone artefact, discerning how these data are tight together

and with other elements is the challenge threw down to researchers.

Biographies of stones are intertwined to help us estimate the mapping of bodies and agencies on different scales, the combination of proofs collected with these tools can be the foundation for interpreting the invisible. In a "*past formed from a turmoil of multiple voices*" (Bray and Pollard 2005, 181) the stone call can be detected by means of digital analytical tools.

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