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## ARCHAEOLOGY: AN OVERVIEW

Liritzis, I<sup>1,2</sup>, Laskaris, N<sup>1,3</sup>, Vafiadou A<sup>1,2</sup>, Karapanagiotis I<sup>4</sup>, Volonakis, P<sup>1,2</sup>,  
Papageorgopoulou, C<sup>5</sup>, Bratitsi, M<sup>1,2</sup>

<sup>1</sup>Laboratory of Archaeometry, Dept of Mediterranean Studies, University of the Aegean, 1 Demokratias Str., Rhodes 85132, Greece.

<sup>2</sup>Laboratory of Environmental Archaeology & Preventive Conservation, Dept of Mediterranean Studies, University of the Aegean, 25<sup>th</sup> March Str., Rhodes 85132, Greece.

<sup>3</sup>University of West Attica, Dept of Industrial Design and Production Engineering, 250 Thivon & P. Ralli Ave, Egaleo, 12244, Greece

<sup>4</sup>University Ecclesiastical Academy of Thessaloniki, 65 N. Plastira str., P.C. 54250, Thessaloniki, Greece

<sup>5</sup>Laboratory of Physical Anthropology, Democritus University of Thrace, Department of History and Ethnology, Panagi Tsaldari 1, Komotini, 69100, Greece

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\*Corresponding author: ([liritzis@rhodes.aegean.gr](mailto:liritzis@rhodes.aegean.gr))

### ABSTRACT

For over 70 years the introduction of natural sciences to archaeology and cultural heritage has been ever growing with multiple contributions to deciphering the past. Novel techniques, applied to almost all material culture, and interdisciplinarity, converged to solving many questions regarding the living and evolution of humans, the environment they lived, the environmental reconstruction, their knowledge about things and the intangible heritage. Archaeometry or archaeological sciences is reviewed regarding major categories of specialties. Characterization and provenancing, dating methods, cyber-archaeology, location of antiquities with geophysical methods, archaeoastronomy, bioarchaeology, geoarchaeology, conservation sciences, and major applications are discussed. Archaeometry is Science at the Service of Human History and Art.

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**Keywords:** archaeological sciences; chronology; cultural heritage; characterization; conservation; provenance; prospection; ancient diet; isotopes; archaeoastronomy, palaeoenvironment, geoarchaeology, cyber-archaeology

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1. INTRODUCTION

Archaeological science, also known as archaeometry, consists of the application of scientific techniques to the analysis of archaeological materials, to assist archaeologists to study and interpret the an-

cient monuments and artifacts, and by extension the human cultures.

The fundamental target of the archeological sciences is the cognizance of past social orders through the investigation by characteristic sciences and new advances of material culture (Jones, 2004; Leute 1987; Gilberto, 2010).

*Archaeology, the sister of History, is completed in the targeted object that is studied through the synergy of the combined natural sciences and the new technologies. No longer as an information on the external peel of an onion-target, but as a continuous peeling in the search for solutions that concern the archaeologist, resulting in the implementation of thousands of applications and the discovery of new methods of problem solving. The knowledge of the roots of humans and the extraction of information from the material culture requires an interdisciplinary and multiscientific investigatory approach, which is realized by archaeometry.*

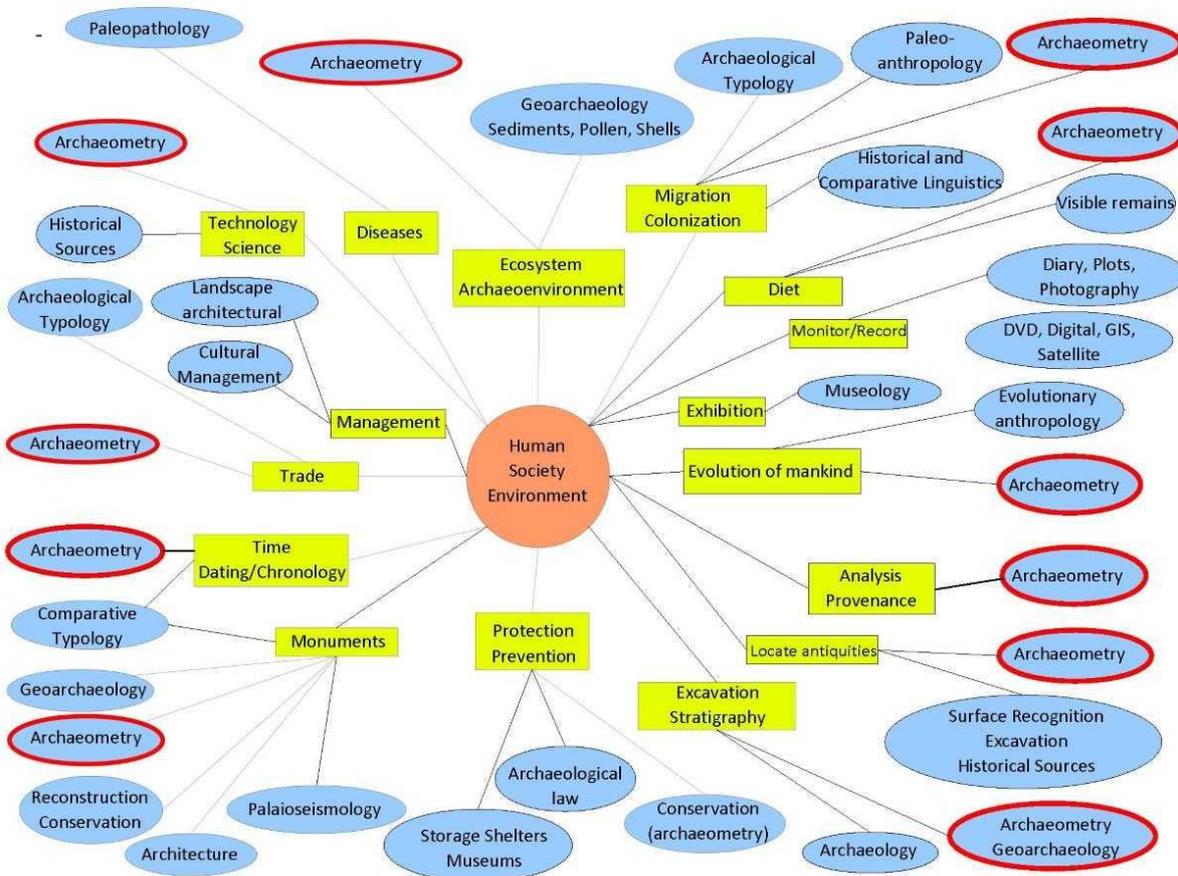


Figure 1 Flow chart that depicts archaeometry along with other scientific fields in ovals (blue) that document the study of the triplet human-society-environment, as questions for the past in rectangular (green), and demonstrates the synergy required for more precise research and emergence of cultures of the past (Liritzis ©)

Contemporary archaeology follows up with being an extremely diverse discipline, encompassing interest groups which focus on distinct periods, regions, theoretical perspectives and methodological techniques.

In spite of the fact that this assorted variety is a positive advancement, emerging problematic issues

due to mis-communication from different components of the discipline (Schiffer, 1996; Brown and Pluciennik, 2001) or improvement of epistemological contentions to coin the advancement, confound the archaeological-archaeometrical connection, and each new term is triggered by archaeological theorists and

archaeological researchers (Edmonds, 1990; Dunnell, 1993).

At any rate, archaeological sciences encompass a wide range of scientific techniques both for the study of heritage objects and historic / prehistoric events. Such techniques are based on basic principles and phenomena of physics (e.g. radioactivity, electricity and magnetism, atomic theory, electromagnetic radiation), chemistry (e.g. diffusion, reactions, melting, affinities), geology (e.g. geomorphology, sedimentology, petrology), geophysics (palaeoclimate, geomagnetic field, atmosphere), astronomy (solar system, celestial mechanics), mathematics (algorithms, statistics etc.), information sciences (Information technology and communication, virtual reality, 3D etc.), to mention major natural sciences fields.

Archaeometry (from *archaeos-* in Greek ancient, and, *-metron* from Greek measure and measurement) etymologically defines the interdisciplinary subject of measurements taken by instrumentations on ancient materials, that is artifacts, monuments and proxies of past environments, to define a parameter inseparably linked to archaeology, history and cultural heritage.

In the interactive and chain by-products frame of homo, society and environment, archaeometry plays an important and indispensable role (Fig.1).

Positive sciences have always been the 'tools' of the study of nature and man. From the tiny atom to the expanding universe, the nature and properties of the cosmic space, even the 'invisible' connection between them, are examined with sophisticated instrumentation and theoretical models of natural sciences.

The *human* (anthropoids) and the *palaeoenvironment*, that have coexisted and have been interacting for over two and a half million years, with at least the current information, and their subsequent evolutionary course, are the roots of modern humans. This marvelous binomial is characterized by: a) its morphological and anatomical evolution; b) its presence and the detailed as possible, cultural development, based on the advancement of tool making, use of fire, search for food, socio-economic structure, (c) migration due to climate change and/or exploration / colonization; (d) reconstruction of the ancient groups and nomads and organized societies, the ecosystem and the ancient-past-environment that lived, (e) the changes in the earth's environment from intrinsic causes (earthquakes, volcanoes, floods, hurricanes, landslides, changing river beds) or astronomical causes (fall meteorites and comets, atmospheric contamination, glaciers, extreme weather conditions, epidemics).

Archaeometry was first introduced as a term and new science at the end of the 1950s in Oxford, Eng-

land, by an archeologist, Professor C.F.C.Hawkes, when he was Professor of European History at Oxford University.

Since the Renaissance, alchemists, natural philosophers, and researchers from the 19th century have been observing and dealing with some natural phenomena that were to be precursors to archaeometry or carried out first analyzes of wet chemistry in archaeological materials. Thus, in the 17th century, the phenomenon of thermoluminescence (TL) was described in an article by the Royal Society of London in 1663 by Robert Boyle describing the effect of light emission from a diamond when heated to the temperature of the human body. This phenomenon in the 1950s was developed in the homonymous method of dating ceramics, burned clay, etc.

In the early 19th century Martin Klaproth, analyzing Roman glasses and coins to determine their technology, Bazzelius analyzed brass objects, and M.Faraday, analyzed polishing in glass jars for the determination of lead. In the middle of the 19th century, Gobel from Estonia tried to analyze brass for provenance with statistical processing, and Darmour from France, non-metallic objects.

In the late 19th century, Helm and the well-known German businessman and a pioneer in the field of archaeology H.Schliemann, analyzed beads from Mycenae, and Richards from Harvard in 1895, made studies of Attic pottery origin. In 1957, Sayre and Dodson carried out measurements of Attic pottery origin with neutron activation. (see, Harbottle, 1982).

A landmark of the recognition and value of the use of natural methods in archaeology is the introduction of the method of dating organic materials (charcoal, bones, shells, rotten wood, plant matter, etc.) with carbon or carbon-14 ( $^{14}\text{C}$ ) invented by the American professor W.Libby, of the Nuclear Sciences Institute in Chicago in the mid-1950s, for which he received the Nobel Prize in chemistry in 1960. (Aitken, 1990).

In the 1950s, the first attempts were made to certify ceramics in Winscosin and Oxford. At the same time, the groundbreaking Research Laboratory of Archeology and History of Art was founded at the University of Oxford by professors Ted Hall and Martin Aitken, who titled the first scientific journal of the new interdisciplinary field, the Archaeometry, which continues to be published.

Although the link between natural sciences and archeology has been 'incidental' by natural scientists working in physics, chemistry, etc., the first PhDs of purely oriented research and applications in archeology have gradually emerged from Universities or research centers. We can say that since 2000s the period that sets the foundation for the forthcoming establishment of the specialty of archaeometry as a *sine*

*qua non* basic learning unit of the curriculum of archaeology.

Interdisciplinarity emerged as a "competent and necessary" tool in the post-war period, and in recent years, for three reasons,

a) the research of a subject requires knowledge of related fields for a more comprehensive and widely applied knowledge, by revising the doctrine of specialization which, as a natural consequence, developed in the late 19th century (with the industrial revolution),

b) It is a major consensus that seemingly different and unrelated scientific fields, which are linked mechanistically (e.g. industrial, synthetic, natural materials) or proportionately (nucleus, electron, atom, cell, molecules, planetary systems, galaxies, radiation, interactions, forces in nature, space-sun-climate-man-earth, etc.) are essentially linked to an interactive mechanism, so that more scientific information in depth and breadth is required to study even one of their components in order to produce integrated and completed models; and,

c) new employment centers are created for the newly formed demanded work (study, prospects, economic development, progress) of the new labor structure (demand-offer), which comes from the technological revolution on a fast rate.

Archaeology is ranked in the field of humanities, arts, social sciences. However, certain methodologies of archaeology go beyond this and becomes 'scientific archaeology' (as an epistemology). It is claimed that this differs from the term 'archaeological science' (the application of specific techniques to archaeological materials), called also archaeometry. Thus, Martínón-Torres and Killick (2017) declare that 'archaeological science' has promoted the development of high-level theory in archaeology, but, Smith (2017) rejects both concepts of archaeological science because neither emphasize falsification or a search for causality.

Whatever terms are given to the coupling of archaeology (but history, culture and arts, too, as sister terms that are omitted) with natural sciences (methods and techniques), the fact is the evidence of proof: the groundbreaking information retrieved from material culture with science, without which archaeology would inhere the sense of doubt.

Archaeometry or archaeological science in a broad sense covers a wide spectrum of partial specialized areas. Under its umbrella several sub-disciplines focus on a specific method or type of material, geographical or chronological focus, or other thematic concern. The eight groups of specializations are:

- *Chronological methods* (relative and absolute methods). Dating by physical and chemical methods.

- *Characterization & Provenance studies*. Physical-chemical techniques and methods for the determination of the chemical content and attribution of the origin of raw materials with appropriate statistical elaboration.
- *Archaeo-geophysical prospection*. Geophysical and remote sensing surveys for detection of ancient buried structures.
- *Archaeoastronomy*. Study of the orientation of monuments related to celestial bodies and inference on ritual practices, religious festivities and determination of time,
- *Biomolecular archaeology*. Ancient DNA and isotopic studies of bones and molecular remains in ancient pots and deduction of used nutritional content, general relationships, migrations.
- *Conservation Sciences*, involving the physical-chemical study of decay processes and the development of new methods of conservation
- *Geoarchaeology – Environmental Archaeology*. Environmental approaches which provide information on past landscapes, palaeoclimates, flora, and fauna.
- *Cyber-archaeology*. The coupling of archaeology, computer science, engineering, and the natural sciences, and it offers 21st century solutions to safeguard the past for future generations and provides digital reconstructions on a virtual environment (Levy and Liss, 2020).

Techniques such as the lithic analysis, palynology, osteoarchaeology, zooarchaeology, palaeoethnobotany, archaeometallurgy, also form sub-disciplines of archaeometry.

Whichever these thematic divisions are, the concept of a perpetually accredited scientific holistic approach (PASHA)<sup>1</sup>, provides current answers to questions arising from contemporary or future problematic issues and / or reassesses past results in the spirit of updating and reassessment. It is a kind of Meta-Archaeology, which involves philosophy, archaeology and natural sciences.

Apart from applications, archaeometry also develops research into new methods and materials to improve errors, increase accuracy and thus reliability.

The important contribution of archaeometry to cultural heritage and archaeology for most of the years of development remained known either to a few open-minded archaeologists or to a narrow group of academia.

<sup>1</sup> Involving theory and practice and covers processual and post-processual approaches for the ever new archaeology.

Eventually the realization of the usefulness of cultural tourism and the realization of valorization of cultural heritage assets, supported by UNESCO and EU projects have placed archaeometry to one of the top priorities for sustainability on national and regional levels (Liritzis 2001; Liritzis and Korka, 2019)

Overcoming the past theoretical approaches, with the archaeological dialogues should continue, however, from now onwards we should consider PA-SHA developed to account for the new diversity and virtual reconstruction, one that integrates micro and macro perspectives – from human life stories (bio-archaeology, a-DNA & isotopes) to their larger social/cultural/environmental framework (travels/interaction/networks/ major genetic shifts/paleoclimates-palaeoenvironments). This way from the archaeometrical data evolves a novel concept; the reconstruction of human past meaning the paleoenvironment and ecosystem, the people and their activities and material and intangible culture. This in turn has led to the introduction of new scientific branches emerging from STEM (science- technology-engineering-mathematics), the STEAM (Psycharis, 2018; Panou, 2018) and recently STEMAC (Liritzis, 2018)

The rapid development of scientific techniques sheds new light for the interpretation of cultural and archaeological growth and evolution (Anderson et al., 2014; Liritzis, 2013; Kintigh et al., 2014).

Today the humanities and archaeology, in particular, needs to engage in discussing the implications of the expanding frontier of knowledge, from archaeogenetics to the diet and mobility of individuals, from demography to sustainability in the long term. We can revisit the museum stores and archaeological sites and select material that reconstruct whole life stories of individuals, their diet, mobility and close family stories, as well as their larger genetic family stories from prehistory until the present. Sedimentological and geoarchaeological data reconstruct the ancient environment. Thus, a new door has been opened to previously hidden absolute knowledge that once again will reduce the amount of qualified guessing and thus both refine and redefine theory and interpretation. Moreover, brings archaeology much closer to the public and public awareness.

The wide interdisciplinary and multi-scientific field of archaeometry can't get exhausted in a few pages and it deserves several books on each sub-discipline.

Here, an overview of archaeometry is given that covers most major issues of the various archaeometric areas along with type of materials, principles of functioning of relevant devices, methods and techniques with selective applications.

## 2. SOME DATING METHODS

On top of the unilateral specialized applications, the holistic approach and double checking methodology should be exerted as a priority issue in any archaeometric result. An example of the multidisciplinary and interdisciplinary methods applied is the Ice man or Otzi; the human body of a Bronze age hunter in the top of Alps.

The ice man 'Otzi' (this name refers to the discovery site in the Ötztal Valley Alps) is a mummy of central European Alps glaciers from the Early Bronze age (3300-3100 B.C.) and has been preserved down to the present day. The body was discovered accidentally by hikers in 1991, together with his clothing and equipment, on the Schnalstal/Val Senales Valley glacier and has been the subject of intensive research. Ötzi and his artefacts have been exhibited at the South Tyrol Museum of Archaeology in Bolzano, Italy since 1998. Following a thorough scientific analysis, the mummy is stored in a specially devised cold cell - a glass vitrine with controlled temperature (-6°C) and humidity (98%) at glacier-like conditions. Ötzi's numerous pieces of equipment and clothing have been painstakingly restored. The magnificent work of conservation, exposition, and reconstruction of this mummy has been initiated by the holistic archaeometric contribution. Techniques applied and materials measured include: a) radiocarbon ( $^{14}\text{C}$ ) dating of clothing, wooden bow, and bone (Bonani, et al., 1994; Kutschera & Rom 2000). b) X Ray fluorescence (XRF) of hair discovering traces of copper and arsenic, implying his involvement in early pyrotechnology of smelting copper, (Artioli et al., 2017), c) X Ray radiography of his whole body discovering the fatal flint arrow in his left back shoulder, and other injuries (Macko et al, 1999; Price & Burton, 2012), d) bio-archaeology used to decoding the Iceman's genetic make-up through aDNA, as well as, carbon, oxygen, nitrogen, strontium isotopic analysis in his teeth and bones proved his southern of Alps (Italy) origin, while he had eat three meals during the last day or so, including a final meal about two hours before he was killed. Methods of Thermal ionization (TIMS), inductively coupled plasma (ICP-MS) and gas mass spectrometry included isotope ratios of  $^{18}\text{O}/^{16}\text{O}$  ( $\delta^{18}\text{O}$ ),  $^{87}\text{Sr}/^{86}\text{Sr}$  and  $^{206}\text{Pb}/^{204}\text{Pb}$ , in order to reveal the Iceman's origin and migration behavior. Analyzed samples include tooth enamel, bones and contents of his intestine, which all represent different ontogenetic (developmental) stages (Macko et al., 1999; O'Sullivan et al 2016; Kutschera & Muller 2003; Muller et al., 2003, Rollo et al., 2002), e) palynology for pollen contained within the foods consumed by Ötzi, along with other palaeopathology evidence, col-

leagues were able to reconstruct his hectic itinerary in the hours before he died and defined the late spring/ early summer death incident as well as the archaeobotanical environment (Bortemschlager and Oegg, 2000), f) virtual reconstruction of the Ice Man Otzi and his equipment, showing how he was equipped for the harsh temperatures of the high mountains and his clothing. The extremely significant information obtained by these archaeometric analyses has had an impact to cultural tourism through increased number of visitors in south Tyrol and especially the Otzi museum (Liritzis & Korca 2019) ([http://www.iceman.it/en/20-years-south-tyrol-museum\\_topics/](http://www.iceman.it/en/20-years-south-tyrol-museum_topics/)).

## 2.1 CHRONOLOGICAL METHODS

Chronological methods are those methodologies by which we can estimate the age of the archaeological object. They also called Dating Methods and are divided into two categories, *relative* and *absolute* dating methods. *Relative methods* of dating refer to the calculation of the age of an archaeological finding through a sequence of comparison with other archaeological finds. *Absolute dating* methods are mainly in the field of archaeometry, since they involve natural sciences so that age can be directly calculated by the artifact (Walker, 2005)

The primary methodology in relative dating methods is the determination of the relative order of events mainly based on the comparison each other. The most know method is the stratigraphic dating were each layer is naturally placed on top of the other and therefore is evidence of a sequence of events. A selected application of relative dating method revealed the last decade is the dating of art works and manuscripts through identification of the chemical composition of pigments and binding media used. Natural and synthetic pigments have a well-known time period usage, therefore, if a pigment is identified on an artwork, through databases an archaeometrist can relatively conclude the period when this work was made (Bell et al., 1997; Caggiani et al., 2016).

Absolute dating methods are based on the physicochemical properties of the materials used by earlier civilizations and also on the interaction of those materials with the environmental conditions (e.g. humidity, cosmic radiation). Absolute dating methods can be used for exact estimation of the age of an artifact.

The dating methods in archaeometry concern those mainly applied to the hominid evolution which spans in the Quaternary, which is also the most recent period of the geological record. Spanning the last 2.5 million years or so of archaeological time and includes Pleistocene and Holo-

cene epochs (West, 1977). However, the latter has been recently replaced with the term *Anthropocene*<sup>2</sup> and spans the last 12-15,000 years.

Such absolute methods are:

### 2.1.1 RADIOCARBON DATING.

Based on the half-life of C-14 isotope is used for dating organic remains (plants, bones and objects made with organic material).

Organisms at the base of the food chain that photosynthesize - for example, plants and algae - or respiration use the carbon in Earth's atmosphere. They have the same ratio of <sup>14</sup>C to <sup>12</sup>C as the atmosphere, and this same ratio is then carried up the food chain all the way to apex predators. But when gas exchange is stopped, be it in a particular part of the body like in deposits in bones and teeth, or when the entire organism dies, the ratio of <sup>12</sup>C to <sup>12</sup>C begins to decrease. The unstable <sup>14</sup>C gradually decays to <sup>12</sup>C at a steady rate (Fig.2). This is the key to radiocarbon dating. Scientists measure the ratio of carbon isotopes to be able to estimate how far back in time a biological sample was active or alive.

C-14 is formed by interaction of cosmic ray spallation products (neutrons) with stable N gas (eq.1).



Radiocarbon subsequently decays by  $\beta$ -decay back to <sup>14</sup>N with a half-life of 5730 years (eq.2).



The radiocarbon dates are determined by measuring the ratio of <sup>14</sup>C to <sup>12</sup>C in a sample, relative to a standard, usually in an accelerator mass spectrometer, AMS (Jull and Burr, 2015). In fact, all living entities absorb this isotope during their life and cease to acquire upon death. In order to investigate stable isotopes from e.g. remains of human and animal bones, a minimal amount of bone sample is required for the analysis. Due to advances in AMS a small sample which can range from 200 milligrams to 1 gram of bone can be used. When archaeological bone material is poorly preserved there may not be enough surviving biological material left for the analysis to be reliable. However, in cases where the bones are well preserved, the isotopic signatures are considered to be representative of the individual specimen (either human or animal) that is being studied (Kromer et al., 2013).

The small bone sample is then treated through a set of chemical procedures, depending on the particular analysis in question. For example, for analysis of carbon and nitrogen stable isotopes, the bone is

<sup>2</sup> From anthropo- from *anthropos* (Ancient Greek: ἄνθρωπος) meaning "human" and -cene from *kainos* (Ancient Greek: καινός) meaning "new" or "recent".

washed in hydrochloric acid (HCl) for an appropriate period of time until the bone sample is ready for the next chemistry steps. These processes are carried out to extract the "pure" bone collagen from additional components that make up bone, such as lipids and proteins. Once the collagen is extracted this is prepared and weighed for analysis in the mass spectrometer (Korlevic et al., 2018).

The mass spectrometer works by measuring the masses and relative concentrations of atoms and molecules. These are compared using standard reference materials that are set by the International Atomic Energy Agency in Vienna. The use of global and national standards as reference material means that isotopic results can be compared across archaeological sites.

For example, the standard could be oxalic acid that represents activity of 1890 wood. Then, <sup>14</sup>C ages are reported as "<sup>14</sup>C years BP", where BP is 1950. However, it is important to remember that the carbon isotopic values of a particular time in the past and place must also be determined in order to un-

derstand the various local processes (environmental and cultural) that are constantly at work.

Radiocarbon dating works as follows (Gopalan, 2017):

- 1) As plants uptake C through photosynthesis, they take on the <sup>14</sup>C activity of the atmosphere.
- 2) Anything that derives from this C will also have atmospheric <sup>14</sup>C activity
- 3) If something stops actively exchanging C (it dies, is buried, etc), that <sup>14</sup>C begins to decay (eq.3) and Fig.2.

$$A = A_0 e^{-\lambda t} \tag{3}$$

where present-day, pre-bomb, <sup>14</sup>C activity = 13.56dpm/g C. Hence, all one needs to know to calculate an age is A<sub>0</sub>, which to first order is 13.56dpm/g. But, small variations (several percent) in atmospheric <sup>14</sup>C in the past lead to dating errors of up to 20%. The Sources of variability are: 1) geomagnetic field strength, 2) solar activity, and, 3) carbon cycle changes.

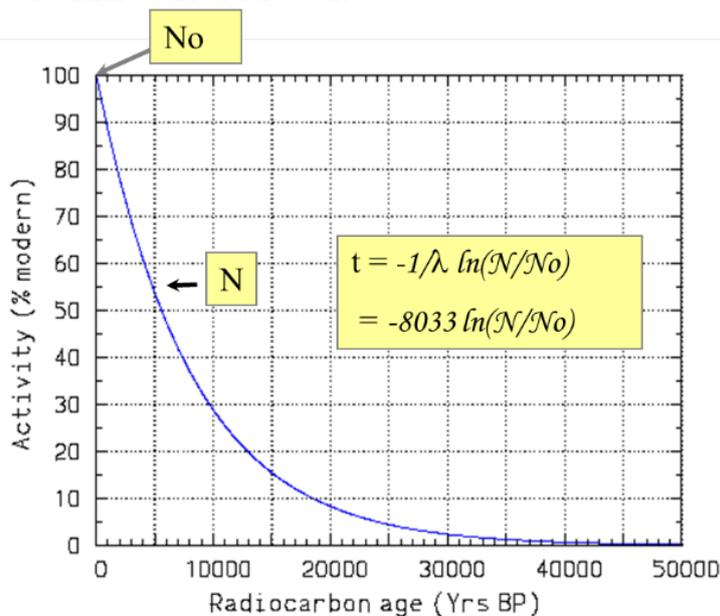


Figure 2. The C-14 activity gradually decays as a function of time with a half-life. The radiocarbon age (t) depends on the initial amount of C-14 (No) and the measured (N) at time (t).

The carbon exchange between atmospheric CO<sub>2</sub> and carbonate at the ocean surface is also subject to fractionation (isotopic fractionation), with <sup>14</sup>C in the atmosphere more likely than <sup>12</sup>C to dissolve in the ocean. The result is an overall increase in the <sup>14</sup>C/<sup>12</sup>C ratio in the ocean of 1.5%, relative to the same ratio in the atmosphere. This rising of <sup>14</sup>C concentration almost exactly cancels out the decrease caused by the upwelling of water (containing old, and hence <sup>14</sup>C depleted, carbon) from the deep ocean, so that direct measurements of <sup>14</sup>C radiation are similar to measurements for the rest of the biosphere. The correction

for isotopic fractionation, is done for all radiocarbon dates and allows safe comparison between results from different parts of the biosphere. This correction gives an apparent age of about 400 years for ocean surface water (<http://www.c14dating.com/corr.html>; Walker, 2005, 2.4.3).

Libby's original exchange reservoir hypothesis assumed that the <sup>14</sup>C/<sup>12</sup>C ratio in the exchange reservoir is constant all over the world, but it has since been discovered that there are several causes of variation in the ratio across the reservoir (Marra, 2019).

Researchers measure the  $^{13}\text{C}/^{12}\text{C}$  ratio, and they use it to correct for “missing”  $^{14}\text{C}$  (eq.4). Thus, the less  $^{13}\text{C}$  a sample has, the less  $^{14}\text{C}$  it has, and so the uncorrected  $^{14}\text{C}$  age will be lower than the calendar age.

$$\delta^{13}\text{C} = \left[ \frac{\left( \frac{^{13}\text{C}/^{12}\text{C}}{\text{spl}} \right) - \left( \frac{^{13}\text{C}/^{12}\text{C}}{\text{std}} \right)}{\left( \frac{^{13}\text{C}/^{12}\text{C}}{\text{std}} \right)} - 1 \right] * 1000 \quad (4)$$

Samples are “normalized” to a  $\Delta^{13}\text{C}$  PDB value of -25‰ and the corrected activity is given in (eq.5).

$$A_{\text{corr}} = A_{\text{meas}} \left[ 1 - \frac{2(25 + \delta^{13}\text{C}_{\text{PDB}})}{1000} \right] \text{dpm/g} \quad (5)$$

By convention the atmospheric radiocarbon anomaly with respect to a standard is defined as  $\Delta^{14}\text{C}$  (eq.6)

$$\Delta^{14}\text{C} = \left[ \frac{\left( \frac{^{14}\text{C}/^{12}\text{C}}{\text{spl}} \right) - \left( \frac{^{14}\text{C}/^{12}\text{C}}{\text{std}} \right)}{\left( \frac{^{14}\text{C}/^{12}\text{C}}{\text{std}} \right)} - 1 \right] * 1000 \quad (6)$$

Calibration curves are constructed to obtain corrected radiocarbon ages taking into account all sources of correction (Reimer et al., 2013).

However, presentation, calibration and interpretation of radiocarbon ages are misleading in some cases, and important technical advances in the pre-treatment of bones and other sample types that have increased dating accuracy have been overlooked.

These considerations have undermined the conclusions drawn about past human dispersals (Turney et al., 2006).

Moreover, identified variations in the carbon 14 cycle due to climatic conditions in our distant past at certain periods of time throws off timelines by as much as 20 years (Manning et al., 2018).

A skullcap found in the Salkhit Valley in northeast Mongolia, the only Pleistocene hominin fossil found in the country, initially described as an individual with possible archaic affinities, but its ancestry has been debated since the discovery. It was C-14 dated by compound-specific radiocarbon dating of hydroxyproline to 34,950–33,900 Cal. BP (at 95% probability), placing the Salkhit individual in the Early Upper Paleolithic period (Deviese et al., 2019)

## 2.1.2 LUMINESCENCE METHODS

**Thermoluminescence (TL).** The TL is based on the assumption that all objects absorb natural environmental radiation into structural energy traps. This energy traps are emptied every time the artifact is heated above a crucial temperature (or exposed to sunlight, see below) (Fig.3). By heating the artifact in controlled conditions and measuring the energy stored in these traps (the trapped electrons escape as light) an archaeometrist can determine the last time this object was heated.

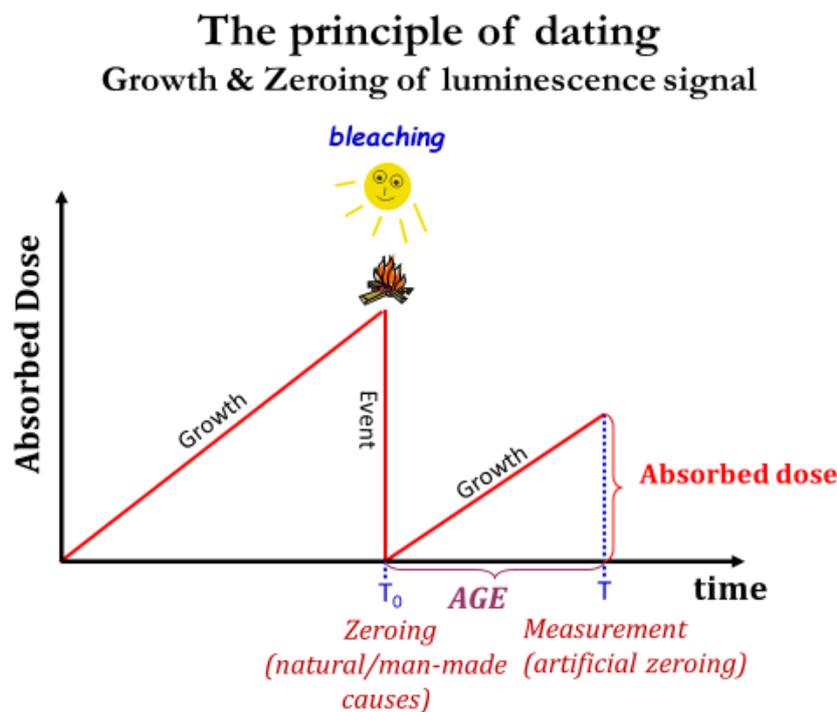


Figure 3. Schematic diagram of the principles for TL/OSL dating.

It is applied to several materials of archaeological or geological origin, such as ceramics, heated sediments and soil, kilns, and then expanded to solar bleached materials, oceans and marine sediments,

flints, rocks (mostly granites, sandstones, calcites). The first application was made in Greek ceramic vessels (Kennedy and Knopff, 1960) and was based on the measurement of the luminescence that is stored in quartz minerals. Since then, the physical properties of the materials to be dated have been studied and various dating techniques have been suggested, based on the sample preparation and the size of the quartz grains used. Ichikawa (1965) and Fleming (1966) studied the behavior and properties of quartz grains. Zimmerman (1967) suggested the fine grain technique, which uses grains of 1-8 $\mu$ m and the accuracy of the technique  $\pm$ 3%, and the technique of large zircon grains (Zimmerman, 1976) with accuracy  $\pm$ 15-20%. Mejdahl (1985) worked on large feldspar grains.

Other techniques include the subtraction technique (Fleming and Stoneham, 1973), with accuracy of  $\pm$ 12% in favourable conditions, and the Pre-Dose technique (Fleming, 1973) with accuracy of  $\pm$ 7%.

When applying any technique, it should be taken into consideration the factors affecting the estimated TL age. Impermanent trap depletion, anomalous fading, light that induces luminescence or non-radioactive induction of thermoluminescence, humidity, non-linear susceptibility change, radon deficiency, changes in  $^{238}\text{U} / ^{230}\text{Th}$  ratio, imbalance of subsidiary nuclei in radioactive U- disequilibrium series (Murray and Aitken, 1982; Liritzis and Danalitsaki 1985; Aitken, 1985; McKeever, 1987; Duller et al., 1997; Greilich et al., 2002; Bailif, 2006).

Concerning the dating techniques for solar bleached artifacts, and in particular the surface luminescence dating of megaliths, the research and the progress in this field of application is presented in chronological order in Liritzis et al., (2013a; see also applications in Greek and Egyptian monuments / artifacts: Liritzis et al., 1997a; Vafiadou, et al., 2007; Liritzis, 2010; Liritzis et al., 2010; Liritzis, et al., 2013b; Liritzis and Vafiadou, 2014; Liritzis et al., 2019).(see Figs.4, 5, 6).



### Temple of Apollo – Delphi

Achaeol. Age: 550 B.C

Age by TL : 470 $\pm$ 200 B.C.

Figure 4. TL dating of Apollo Temple southern polygonal wall, Delphi. Test of reliability. (Liritzis et al., 1997a).



Strofilas- Island of Andros

Arch. Age: 2800-4200 B.C  
 Age by TL: 3570±400 B.C.  
 (TL of Calcites)



Canal of Eupalinos -Samos

Arch. Age: 530 B.C.  
 Age by TL: 570±300 BC  
 (TL of Calcites)

Figure 5. (left) OSL dating of engraved with ships carved cobbles in fortified wall of prehistoric Strofilas, Andros (Liritzis, 2010) and (right) OSL dating of construction of 1036 meters Eupalinos aqueduct, Heraeon, Samos Island, Greece (Liritzis, 2000).

Stencil art in the Kimberley region Australia. The superimposed Bradshaw painting was overlying by Wasp Nest, which was dated by OSL giving a min 17,000 BP for art-work.



5

Figure 6. The OSL dating of single grain quartz of mud-wasps overlying rock art in Kimberley Australia. The quartz grains extracted from the whole nest minus the portion left on the rock wall. (Aubert, 2012)

The OSL dating method applied to mud-wasp nests in Kimberley rock art paintings was an innovative and promising avenue for rock art dating. However, in order to accurately date rock art with this approach, the relationship between the OSL dated

quartz grains and the art needs to be clearly demonstrated (Aubert, 2012).

**Optical Stimulated Luminescence (OSL).** It is based on the same assumption as TL but with the difference that the energy traps are emptied by sunlight. Therefore, through optical stimulation of the

object (with a laser of specific wavelength), and by measuring the trapped energy the date of burial can be calculated. In this method, it is also assumed that the artifact is buried since last use, and in megalithic monuments the side of the stone is protected from the sunlight. This is the reason why this method demands the sampling procedure to be under dark conditions.

The materials that are dated with this TL can be dated by OSL as well. The techniques that have been applied concern measurements on a single aliquot or a single grain (Liritzis et al., 2013a), (Single Aliquot Additive Dose, Liritzis et al., 1997b), (Single Aliquot Regeneration Added dose SARA, Mejdahl and Botter-Jensen, 1994, 1997), Single Aliquot Regeneration (SAR) (Murray and Wintle, 2000), to both heated and solar bleached quartz and feldspar samples. In order to separate the luminescence components from mixed samples, quartz/feldspar, the method of Pulsed OSL is used (Denby et al., 2006).

Special reference is made to the case of feldspar dating. Since the sensitivity of quartz samples is often low, attempts have been made to use the feldspar signal to date the sample. In order to compensate the loss of luminescence signal (anomalous fading), several methods have been developed to enable corrections of the fading component. Recent applications, the IRSL signal from multi-grain sizes sample, range from 90 to 250  $\mu\text{m}$ , using an isochron (Li et al., 2007, 2008), b) the IRSL measurements at elevated temperatures, e.g. 290  $^{\circ}\text{C}$ , after the IR signal is read out at 50  $^{\circ}\text{C}$  (Ankjaergaard et al. 2010; Buylaert et al. 2009; Jain et al. 2011; see also, Liritzis et al., 2013a and references therein). The post-IR luminescence signal measured at elevated temperature has a smaller fading rate than the signal measured at 50  $^{\circ}\text{C}$ . Finally, Krbetschek and Erfurt (2003) studied the radiofluorescence signal, which monitors an IR-signal around 865 nm that is emitted by electron capture at luminescence traps during ionizing laboratory irradiation. This way, radiofluorescence is not influenced by the possible problems related to lifetimes (fading), electron competition and other features concerning luminescence recombination centres, but refers only to the luminescence traps (Buckland et al., 2019; Prasad et al., 2017).

For the reliability of the dating protocols two tests have been developed and applied at every measurement. First, the Recycling point. The repetition at the end of the procedure of the first and the lowest dose of regeneration cycle. In order to correct the sensitivity changes after continuous irradiation, the corrected signal value should be approximately the same as the first irradiation to be reliable. The second test concerns the measurement of the corrected signal after exposure of the tablet to zero dose. This

should of course be zero, but in reality there is a very small signal. Recuperation is associated with the transfer of electrons due to preheating by traps that are difficult to empty by visual stimulation in light centers (Aitken, 1985).

The OSL method of sediments helped to date past earthquakes (Fattahi, 2009) identify the climatic response of glaciofluvial systems in South Island, New Zealand (Rowan et al., 2012) and to establish a chronology for the stratigraphic sequences for the Pleistocene deposits and associated remains in Denisova Cave. Thus, the reconstruction of the environmental context of hominin occupation of the Altai region of Siberia (which was inhabited for parts of the Pleistocene by at least two groups of archaic hominins; the Denisovans and Neanderthals) from around 300,000 to 20,000 years ago was made possible (Jacobs et al., 2019).

Several applications in ocean, fluvial and lake sediments have been published as well as modern studies (Mejdahl and Christiansen, 1994; Wintle and Huntley, 1980; Huntley et al., 1985; Kale et al., 2000; Jacobs, 2008; del Valle et al., 2016; Tsukamoto et al., 2017; Chamberlain and Wallinga, 2019; Smedley et al., 2019; Sanderson and Kinnaird, 2019)

In another recent application, the loess-paleosol profiles at Krems in central Europe (Lower Austria) the OSL dating and sedimentological analyses are reported for new profiles. These sedimentary profiles are well known for the impressive output of Upper Palaeolithic remains as well as for their paleoclimate potential as terrestrial archives (Groza et al., 2019)

Other interesting case studies concern the dating of a fossil mud-wasp nests in Northern Australia (Yoshida et al., 2003), of the Chiemgau meteorite impact in Bavaria (Liritzis et al., 2010), of sulfates from Martian meteorites (O'Connor et al., 2011), of portable paintings, by measuring ground layer materials (Polymeris et al., 2013), the sea levels reconstruction in Antarctica using optically stimulated luminescence of cobble surfaces (Simms et al., 2011), and a rock-art from India (Liritzis et al., 2018).

Apart from the usual diodes used for the application of luminescent dating techniques, Ankjaergaard et al., (2016), used the violet stimulated luminescence (VSL) signal (405nm) to date at the Luochuan loess type section in China, soil samples from loess and paleosol layers. As a result, it is proposed by the authors the potential to extend the application of the quartz dating to fully cover Quaternary.

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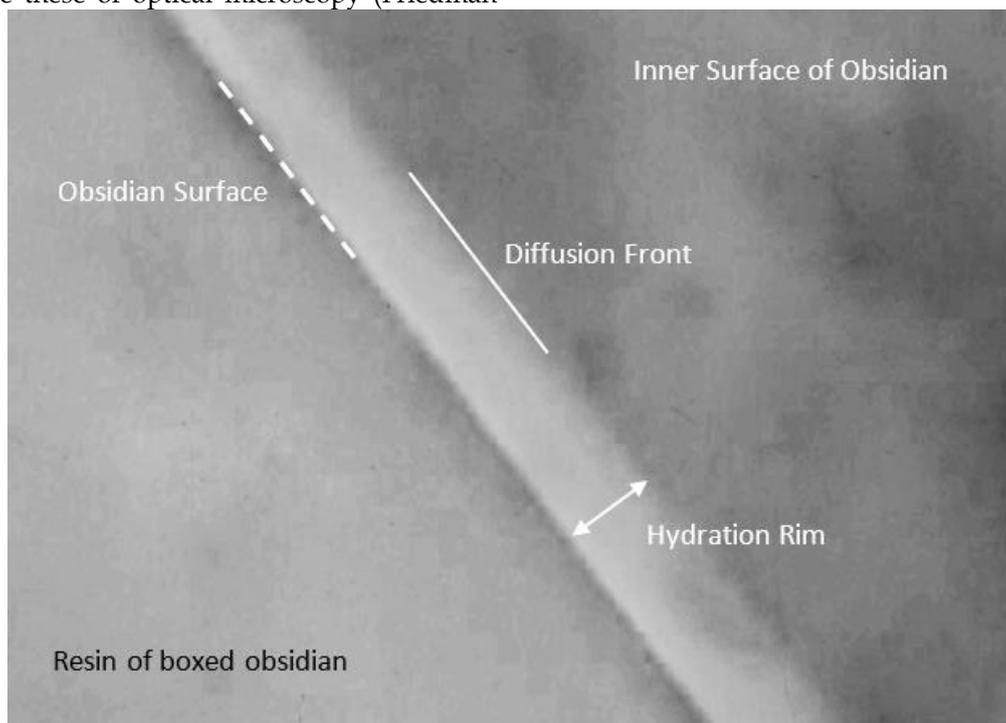
region of Siberia (which was inhabited for parts of the Pleistocene by at least two groups of archaic hominins; the Denisovans and Neanderthals) from around 300,000 to 20,000 years ago was made possible (Jacobs et al., 2019; Reich et al., 2010).

### 2.1.3 OBSIDIAN HYDRATION DATING

Obsidian, as a type of volcanic glass is rare and can be found only in special sources around the Globe. Its formed due to the very fast cooling of lava at volcano slopes (Raymond, 2002).

The dating method concerns the time that the stone tool was made from obsidian. It is based on the phenomenon of hydration on which obsidian absorbs molecular water from the burial environment forming a hydration rim (Friedman and Smith 1960; Liritzis, 2014). The amount of water is measured, the diffusion coefficient is calculated and in the end the age of the artifact in years before present (Friedman and Smith, 1960; Liritzis 2006; Liritzis and Laskaris, 2012). Obsidian's hydration rim measurement is currently based on several different techniques with most usable these of optical microscopy (Friedman

and Smith 1960, Garvey et al., 2016, Nakazawa, 2018), Secondary Ion Mass Spectroscopy (Liritzis 2014, Liritzis and Laskaris 2012, Riciputi 2002) and IR-PAS (Stevenson and Novak 2011, Stevenson et al., 2013). These three methods compared each other shown that optical microscopy is less accurate than SIMS method (Anovitz et al., 1999; Stevenson, 2001, Nakazawa 2018). Higher precision has the Infrared-Photoacoustic Spectroscopy (IR-PAS) method, correlating the water peak's height with rim width (Stevenson 2001, 2002). The most accurate measurement is by using SIMS with  $\pm 0,005\mu\text{m}$  error (Liritzis, 2014). The hydration layer by optical microscope as it was used in early days of the method Friedman and Smith (1960) is shown in Fig. 7. while in early 2000's two different research groups (Liritzis and Diakostamatiou 2002; Riciputi et al., 2002) suggest the use of Secondary Ion Mass Spectroscopy (SIMS) for the measurement of diffused water's profile in the outer surface layers of an obsidian tool is shown in Fig.8.



*Figure 7. Hydration rim under microscope.*

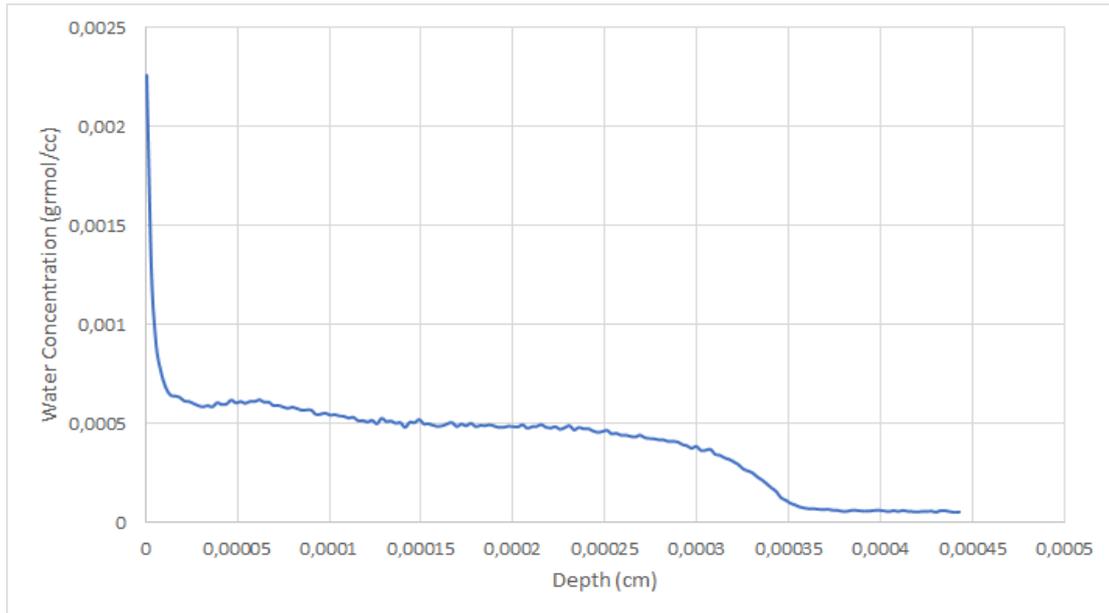


Figure 8. SIMS H<sup>+</sup> sigmoid profile for sample from Youra cave (Greece) (YR-3) (Liritzis & Laskaris ©). Water represented by hydrogen atoms/cc, and depth in cm (=10<sup>4</sup> μm) from the surface into the obsidian body, here diffusion is reaching a depth of 3.5 microns.

The calculation of the absolute age in the obsidian hydration dating method has three major different approaches. First approach was initiated by Friedman and Smith (1960) and it is based on the empirical equation  $X=(kt)^{1/2}$ . Where  $x$  is the hydration rim width in microns (μm),  $t$  is time and  $k$  the hydration rate. Second approach is based on modeling the hydration profile by SIMS method (Anovitz et al., 1999; Liritzis 2006; Liritzis and Laskaris, 2009, 2011). Third

method was initiated by Stevenson and colleagues (Stevenson and Novak, 2011, 2012; Stevenson and Rogers, 2016) and it is based on the high precision correlation of IRPAS data with SIMS measurements. The authors propose the use of water content measurement with IRPAS (OH, H<sub>2</sub>O) for the estimation of hydration rim width and of Arrhenius constant and therefore the archaeological hydration rate, which both leading to an IRPAS - age correlation (Fig.9).

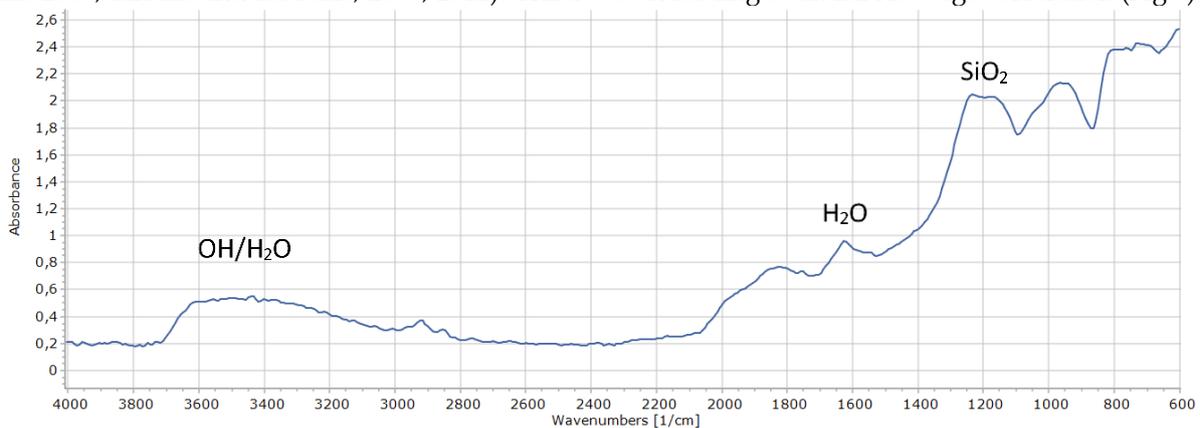


Figure 9. IRPAS spectrum of IR absorbance versus wavelength. Note the prominent water peaks at ~1580 and 3500 cm<sup>-1</sup>. Sample comes from Sarakinos cave, Central Greece (SAR-9) (Liritzis, Laskaris, Stevenson ©).

In several researches, obsidian hydration dating was applied in case studies of artifacts from archaeological sites with obsidian artifacts from known sources and compared to other independent age calculations (Riciputi et al., 2002; Stevenson, Abdelrehim, Novak, 2004; Stevenson, 2011; Liritzis and Laskaris, 2011; Liritzis, 2010, 2014).

As Liritzis and Laskaris (2011) have shown obsidian hydration dating using SIMS as measuring technique along with the Saturated Surface approach (Liritzis, 2004; Liritzis and Laskaris 2011; 2012) has a time range limitation from few years up to 30,000 years BP (Liritzis, 2014); the maximum span relates to the climatic conditions (e.g. for low diffusion

rates) and the peeling effect of supersaturated external surface layers.

One of the more visible tools in the Rapa Nui archaeological record is a large flake modified to form a broad blade and tang. Referred to as Mata'a, these implements have conventionally been interpreted as "spear points", or weapons of war, which are proposed to have been made in large numbers during a period of internal conflict in the late seventeenth century. Obsidian hydration dating of 63 Mata'a fragments from the southern coast of Rapa Nui indicates that their usage peaked in the sixteenth century, declined in frequency afterwards, and briefly spiked in the early AD 1700s (Stevenson and Williams, 2018)

The result of usage or intrusion of small obsidian artifacts from overlying Neolithic layers, is a unique archaeological evidence regarding the presence of obsidian in levels that antedate the food production.

Notable examples of the significance of obsidian hydration dating as an absolute dating method is that of enlighten about the late Pleistocene/Early Holocene Aegean seafaring (Laskaris et al., 2011; Simons, 2012), the dating of obsidian tools in west-central Argentina (Garvey et al 2016) the and the investigation of site integrity of the Early Jomon midden in the Holocene site of Ocharasenai (OCH), Hokkaido, northern Japan (Nakazawa, 2016).

A novel obsidian hydration dating method (SIMS-SS) has been introduced (Liritzis and Diakostamatiou, 2002; Liritzis, 2014; <http://dms.aegean.gr/sims-ss/method.html>). This dating project offered new results of absolute dating concordant with the excavation data, and provided new light on the Late Pleistocene/Early Holocene exploitation of obsidian sources on the island of Melos in the Cyclades reporting dates c. 13th millennium - end of 10th millennium B.P. (Laskaris et al., 2011). Examples of SIMS-SS dating of obsidian tools from various parts of the World are found in Liritzis & Laskaris (2012).

### 2.1.4 RADIOACTIVE DATING

In this category of absolute dating, all other radioactive/decay dating techniques are included. Those techniques are based on well-known radioactive decay rates of several isotopes. This category includes, Uranium Series decay dating, method that dates bones (Holen et al., 2017) and cave deposits (stalactites, stalagmites, travertines), corals (Cheng et al., 2000, Cobb et al., 2003 Yehudai et al., 2017), Potassium - Argon and Argon-Argon dating<sup>3</sup>, applied to rocks and materials and the isotopes are measured with spectroscopic techniques (Schmitt and Vazquez., 2018; McDougall and Harrison, 1999).

<sup>3</sup><https://geoinfo.nmt.edu/labs/argon/methods/home.html>

## 3. CHARACTERIZATION & PROVENANCE STUDIES

As the human race evolves, the tools and techniques used are also evolving. Thus, in the beginning, humans use primary source materials found in the immediate surroundings and, as the evolution runs, they search for new materials with better properties at greater geographical distances. Thus, another archaeometry's field of study focuses on the characterization of an artifact and evaluation of its provenance, to answer crucial questions posed by archaeologist / anthropologist on the trade networks or populations mobility.

Archaeomaterials analysed include: *inorganic* (faience, stone, ceramic, glass, sediment, pigment, metals, fossils), and, *organic* (bone, skin, wood, paper, textile, plant remains).

Main provenance studies focus on:

### 3.1 OBSIDIAN PROVENANCE STUDIES.

Obsidian origin is of high importance for defining the source from which an obsidian tool was quarried because the archaeologist can conclude on the *chaines operatoire* and the way this obsidian traveled from its source to the archaeological site. For the determination and grouping of the obsidian sources, several techniques have been proposed such as the bulk physical properties with rarely good results (Gopher 1983), the hydration data (Stevenson et al 2002) and more accurately, the elementary composition (Williams et al., 2012, Glascock and Giesso, 2012; Shackley, 1998; Tykot, 2002; Liritzis & Stevenson, 2012). The elemental analysis approach for the discrimination of obsidian sources is based on the fact that every source has different concentration of major, minor and trace elements, in a homogeneous distribution. For the determination of these quantities, especially those of trace-elements, several procedures, most of them destructive, have been used, such as, neutron activation analysis, electron microprobe, ICP-MS/AES (Tykot, 2002; Bellot-Gurlet et al., 2002; Pereira et al., 2001). On the other hand, archaeological obsidian its necessary to be studied non-destructively and therefore X-ray fluorescence offer good alternatives (Liritzis, 2007). Yet several factors need particular attention for accuracy and precision, including the correct calibration of the portable instrument, the surface roughness, the spectral resolution of measured peaks.

An obsidian provenance analysis using energy-dispersive X-ray fluorescence of Neolithic tool assemblages from the early to mid-sixth millennium B.C. settlement at Göytepe, Azerbaijan revealed a previously unknown diachronic change in obsidian use in the region, suggesting the occurrence of sig-

nificant socioeconomic changes during the Late Neolithic of the southern Caucasus (Nishiaki et al., 2019).

Other glaring examples are: a) the discovery that the obsidian in the Neolithic layer of the excavation at Mandalo village at Giannitsa, Greece was at the same time from the sources of Milos and Carpathian (Kilikoglou et al., 1996), b) the establishment of the early Holocene seafaring in the Aegean, based on obsidian findings in mainland Greece (Laskaris et al., 2011; Simons, 2012), c) the obsidian circulation in the early Holocene Aegean (Carter et al., 2018), d) the earliest occupation of Central Aegean (Naxos Island) and the Homo Sapiens behavior (Carter, 2019) and e) the long-distance stone transport on middle stone Age for Olorgesailie basin in Kenya (Brooks et al., 2018). Several novel approaches and world applications are found in the collective New Mexico Press Book edited by Liritzis & Stevenson (2012).

### 3.2 ORE PROVENANCE STUDIES

Characterization is based on two different approaches. First is the approach of lead isotopes. Analyses of lead (Pb) isotopes such as Pb-206, 207 and 208 assumes that lead has different isotopic composition among ore deposits. On the other hand, the other approach supports the idea that provenance studies of ancient metal artifacts should be based on geological, mineralogical and geochemical information (Pollard, 2018; Gomes et al., 2018). As for gold or silver artifacts this approach is based on

the trace element existence percentage (e.g. for ochres, MacDonald et al., 2018; Baron et al., 2019.)

The variations in isotope ratios (Pb etc.) can be useful in an archaeological investigation of potential geological sources of raw materials. The polychrome glazes from the Processional Way and the Ishtar Gate of Babylon have been studied, as they are among the best preserved from antiquity, and yet, little is known about the logistical choices involved in their production. In Asia Minor - Anatolia (today Turkey) ore deposits are thus tentatively proposed as possible sources of the metal oxide colorants used for the investigated polychrome glazes of three Neo-Babylonian faunal reliefs that are now part of the collection of the Ny Carlsberg Glyptotek, Copenhagen. This work is consistent with archaeological evidence and ancient texts on trade in metals, (Rodler et al., 2019)

On the other hand, glass, first appears in the 3rd or 2nd millennium B.C. in the wider area of Mesopotamia. In the Aegean it appears in the Mycenaean palaces around 1500 B.C. It remains popular until the end of antiquity (Fig. 10). Used in everyday life, mounted and in graves and offered as a votive offering (Janssens, 2013).

It is often hard to discriminate between glasses made at different factory sites by using chemical analysis alone. However, this is not necessarily a means of provenancing them unambiguously because glass of slightly different compositions may have been fused using different proportions of the same raw materials (Fig. 11).



«The apples appear more beautiful through a glass vase....»

Lucius Annaeus Seneca, *Naturales Quaestiones*, I.6

Figure 10. Glass was popular in antiquity.

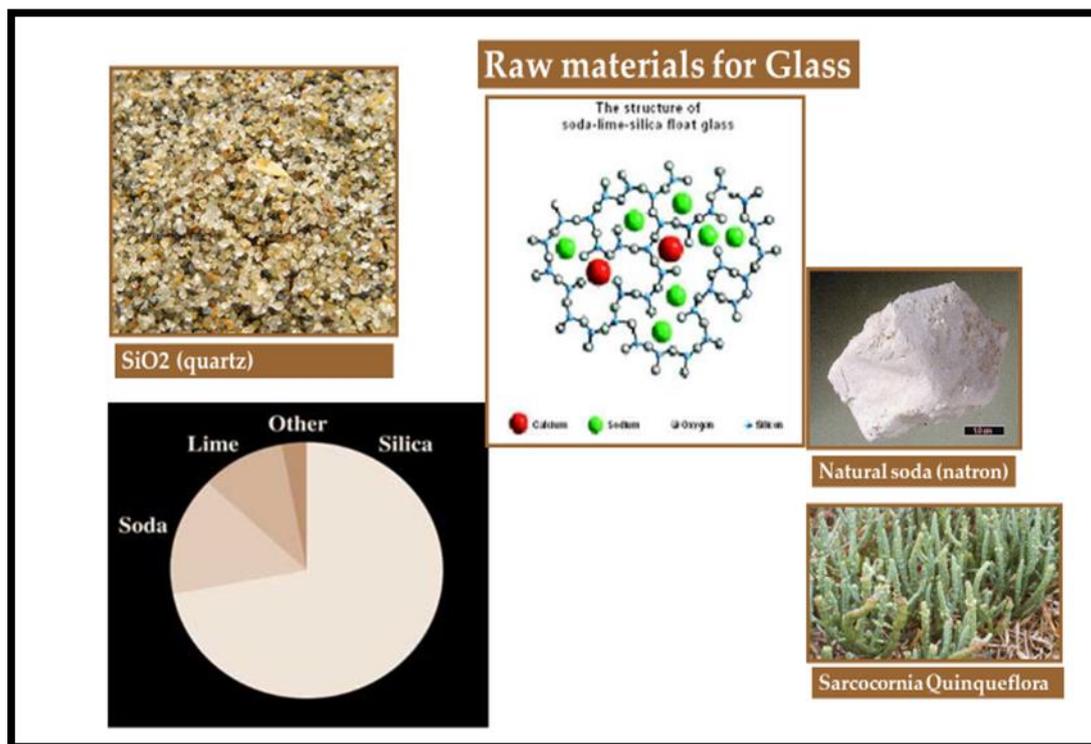


Figure 11. Raw materials for glass.

Metal artifacts (iron, copper, lead etc.) provenance is based on chemical characterization and isotopic analysis and proper statistical processing (Żabiński *et al.*, 2020; Vogl *et al.*, 2019; Rose *et al.*, 2019; Berger *et al.*, 2019; Nørgaard *et al.*, 2017; Dissler *et al.*, 2017).

The determination of oxygen (O), strontium (Sr) and lead (Pb) isotopes can provide the possibility of linking the geological sources of the glass raw materials to the production sites on which the glass was fused. The possible isotope contributions made to the raw materials which are thought to have been used in the manufacture of plant ash and natron glasses found at 8th–9th century al-Raqqā, Syria has been reported. The isotopic data from al-Raqqā are compared with published results from other Middle Eastern and German glasses. It has been shown that strontium isotopes in particular, provide a reliable means of distinguishing between the sources of plant ash glass raw materials and that oxygen and lead isotope signatures are less discriminatory (Henderson *et al.*, 2005).

Sound examples of how glass provenance studies affect the study of human past, also, include: a) the elucidation of late Bronze Age glass production in Egypt where the authors provide evidences for primary production of glass in Egypt (Rehren and Pusch 2005; Jackson 2005), b) the multimethod (SEM, XRF, etc) identification of the glass routes of Mycenaean glass found in Late Bronze Age/Mycenaean sites in NE Peloponnese, Greece (Zacharias *et al.*, 2018), and, c) the technological examination of glass

tesserae from Hagia Sophia in Constantinopolis which found to be originated from Levant and (one case) from Byzantine-Syrian origin (Moropoulou *et al.*, 2016).

### 3.3 CHEMICAL PROVENANCE OF CERAMICS

The analytical techniques used for the characterization and origin of raw clay sources for ceramics is a known approach in archaeological sciences (Maggetti, 2001; Munita *et al.*, 2003; Marketou *et al.*, 2006; Mantzourani and Liritzis 2006; Papageorgiou and Liritzis 2007; Liritzis and Zacharias 2011; Gajić-Kvašček *et al.*, 2012; Epossi Ntah *et al.*, 2017; Javanshah, 2018). It involves chemical and mineralogical analysis by physical methods (e.g. desktop or portable XRF, thin sections and PLM, XRD, optical microscopy, polarized light microscopy (PLM), PIXE, ICP, neutron activation analysis (NAA) (Glascock and Neff, 2003) and multivariate statistical methods, such as, cluster analysis or dendrograms with various clustering algorithms, principal component analysis (PCA) biplots and 3D plots (Mantzourani & Liritzis 2006; Baxter, 2015). Several case studies offer clues concerning the extension of the territory exploited by ancient people, evaluate many aspects of the manufacturing process which comprises all operational acts that transform raw material to the final product (*chaîne opératoire*; Roux, 2016) and ultimately contribute to the knowledge of long-distance circulation and ex-

change of raw materials and goods. Indeed, reconstructing mobility strategies is a major goal of researchers interested in prehistoric societies and the use of geochemical source characterization of ceramics found at sites in a region offers a way to reconstruct the procurement range – local in close proximity, travelled long distances or trade exchange - by prehistoric groups to obtain resources (Mcalister and Allen, 2017). Either way it reflects major issues concerning their technological development, independency and identity of settlements compared to other major centers. Fired clay products (pottery, vases, architectural parts, kilns) are investigated for their raw clay provenance (including mixing ingredients), but in particular pottery, due to its remarkable storage properties, was a vital item used in the food activities of everyday life. Yet ceramic artifacts occasionally may have had a valuable intangible meaning (e.g. figurines) as well. Not only these uses, but aesthetic qualities, too, were frequently used by people. Thus, ceramics are the preferred materials in provenance studies as their physico-chemical properties are most often different at a major, minor but also trace element level, because of their mode of manufacture from characteristic clay sources. Because, once produced, ceramics are virtually indestructible, they are found in quantity at the majority of archaeological sites dating from the Neolithic period onwards. Their study in terms of typology has, therefore, always been central to the archaeological interpretation of a site, region and period, and in consequence, ceramics have been a major focus of archaeometric or archaeological science studies from their beginnings in the 1950s (Tite, 2008). However, according to Kolb (2014), unlike other type of materials, clay, and thereby ceramics, because of its mineralogical complexity it is difficult to determine its source. Different and heterogeneous clays from different sources could be mixed. The firing conditions and temperatures are neither standard nor stable and knowing that at each stage of firing the mineralogical composition of the ceramic changes, make it difficult to define the clay source accurately. An effort to determine whether the raw material is a mixture of local clays is by collecting soil from different sites the area near the excavation site and from the same layer with the pottery. Then briquettes are being prepared, from each collected site and mixed, and fired at different temperatures. Finally, the chemical analysis of the briquettes is compared with the that of the ceramics, If there is a consistency regarding chemistry, mineralogy and thermal properties (Hein et al., 2009; Garcia-Heras et al., 2001; Bronitsky and Hamer, 1986; Liritzis et al., 2019).

Reference clays for Moroccan pottery were established based on the clay mineralogy and chemical

composition. This study helped to answer some archaeological questions concerning possible sourcing areas for archaeological ceramics, to determine techniques for the production of artefacts, and then to interpret cultural influences (El Ouahabi et al 2019).

In a recent study typologically selected ceramics from the Late Mycenaean (Helladic) settlement of Kastrouli central Greece have been analysed by an electron probe microanalyzer (EPMA) equipped with four wavelength-dispersive spectrometers and one energy-dispersive spectrometer. The aim to unravel the “ceramo-genetic” processes was possible by integrating optical microscopy, and EPMA (with SEM capabilities) following a specific protocol narrowing down to  $\mu\text{m}$  scale. A detailed characterization for their texture and major element chemistry was made, including collection X-ray maps for different elements showing the zonation of minerals in relation to the adjacent matrix. Here the temperature range was estimated based on the observed mineral assemblage, texture and chemistry, with favorable firing temperature maxima of 900–1000°C (Baziotis et al., 2019). From same site a satisfactory attempt was made to characterization and provenance ceramics from RGB chromatic index (Bratitsi et al., 2018).

Other, interesting examples of how ceramic provenance include: a) the use of analytical techniques in order to trace the provenance minerals in Roman lead-glazed ceramics (Medeghini et al., 2020), b) a contribution to the identification of pottery production in Late Classic-Hellenistic Northern Greece (Santos et al., 2020), and, c) the elucidation of how ceramic exchange is related to political centralization in the Valley of Oaxaca, Mexico, 700BCE-200CE (Sherman et al., 2019). More examples are included in the recent work about the archaeometry of ceramic materials (Eramo and Mangone, 2019).

### 3.3 MAINSTREAM INSTRUMENTATION FOR CHARACTERIZATION

As for the characterization studies the dominating techniques to gain information on sample composition are:

- **XRF (X-Ray Fluorescence).** The X-Rays Fluorescence is based on the interaction of X-Rays with a substance. Inorganic materials when exposed to primary x-rays they produce secondary or fluorescence x-rays which are analyzed. All elements except hydrogen and helium can produce secondary x-rays. Also, primary x-rays are from several sources such as a tube, synchrotron or radioactive isotope (mainly Am-241). These secondary x-rays are unique for each element, therefore the XRF spectrum has several peaks. The intensity of each peak is correlated to the amount of the element presence in the artifact. The

resolution of the instrument depends on its source. Reference materials are used to calibrate the instrument and to check and control the reliability of the results. XRF analysis is non-destructive non-invasive method and can be applied to inorganic compounds (Liritzis and Zacharias, 2010). The portable apparatus allows the user to perform analysis in situ at the excavation site or in a museum, at artifacts that cannot be removed from their storage space (Liritzis *et al.*, 2018). In order to analyze a small detail on an artifact, a  $\mu$ XRF is used (Uhlir *et al.*, 2008), and in the case of areas in paintings or manuscripts a macroXRF. MacroXRF allows the spatial distribution of chemical elements to be determined over large areas, allowing researchers to infer the presence of certain pigments or mixtures throughout the surface of a work of art (Ricciardi *et al.*, 2016).

- **NAA (Neutron Activation Analysis)**. NAA is used to detect and quantify major, minor and trace elements in a sample. NAA has an extensive use in provenance studies of inorganic archaeological material such as lithics, ceramic and glass. The detection limit of NAA is parts per million. (Hamidatou, 2019a). NAA has an extensive use in provenance studies of inorganic archaeological material such as lithics, ceramic and glass. (Sekimoto *et al.*, 2016, Bland *et al.*, 2017, Bode, 2017, Suda *et al.*, 2018, Maroti *et al.*, 2018, Hamidatou, 2019b)

- **ICP-MS (Inductive Coupled Plasma - Mass Spectrometry)**. The sample is atomized and ionized at very high temperatures. In difference with other techniques of analysis, ICP-MS analyze the mass of ions of the sample rather than the emissions of excited atoms or ions. It is quite efficient in producing positive ions and therefore is used in elemental isotopes discrimination and very low detection limits (Thomas, 2013). The method is applied in several materials, ceramics, glass, obsidian, metallic objects, bones, pigments, art objects (Dussubieux *et al.*, 2016; Gehres and Querré, 2018; Maurer *et al.*, 2019; Zipkin *et al.*, 2017).

- **FTIR (Fourier Transform - Infrared Spectroscopy)**. This FTIR phenomenon is due to the natural vibration that atoms have under covalent bonds. The molecule stays still but the atoms change relative to each other. If a molecule been exposed to Infrared radiation with frequency same as the natural vibrational, energy is absorbed. In fact, this type of analysis involves the interaction of radiation with molecular fundamental vibrations, therefore is included in the category of vibrational spectroscopy analytical techniques. The sample is irradiated with an infrared beam (near-IR, mid-IR), molecules start to absorb this radiation and change molecular vibrational energy levels. As a technique can be used for simple identification of molecular compounds or even for a

full qualitative and quantitative analysis. Notable examples of use are for pigment identification (Silva *et al.*, 2006), historic paper analysis (Zotti Ferroni Calvini 2008), evaluation of Paleolithic artefacts (Carciumaru, 2012) and other archaeological materials (Ganetsos *et al.*, 2010).

- **Raman Spectroscopy**. The Raman Spectroscopy is a type of vibrational spectroscopy based on the inelastic scattering of monochromatic light mainly from monochromatic lasers. As monochromatic laser beam spots a sample it interacts with molecules of the sample and be scattered shifted. This Raman Shift is unique for every type of molecules resulting to a fingerprint spectrum of each chemical composition. It differs from FTIR in the way energy is transferred to molecules. Here, the sample is irradiated with a laser beam of visual or near IR, the incident photon has higher energy than of the vibrational state of the molecule and therefore a portion of the photon energy is absorbed leading to a molecular vibration and the remaining energy is scattered as a new photon with reduced frequency. As FTIR, Raman spectroscopy can be used for qualitative and quantitative analysis. Both Raman and FTIR should be used complementary for a full characterization of the vibrational modes of a molecule. Raman Spectroscopy has several applications in archaeology such as for the Rock art analysis, pigment identification, lithics, textiles, resins and organic residues (Smith and Clark 2004).

#### 4. ARCHAEO-GEOPHYSICAL PROSPECTION

Modern archaeologists do not excavate simply to accumulate data, but to solve problems. Hence, they are not content with waiting for some random discovery (e.g. making a road, erecting a building), or digging monuments simply because they have become visible. Instead, they focus on trying to retrieve sufficient evidence, wherever they are. Thus, archaeologists are taking advantage of the available detection techniques, which were developed at an advanced level in the search for oil, water, stratigraphic or military purposes. As archaeologists learn to share the great adventure of basic research, they began to appreciate geophysical instruments and observation techniques for locating buried antiquities, such as the proton magnetometer, the electrometer, the georadar (GPR), the soundings the side scanner; not only as useful accessories but as tools of fundamental importance (Deiana *et al.*, 2018; Clark, 1996).

In general, the discovery of archaeological sites from the ground is achieved, from:

- (a) written sources,
- (b) (accidental) rescue archeology, and
- (c) topographical survey

The search for and excavation of sites the execution the surface study of a site is achieved rapidly and efficiently by the detection of the subsoil by non-destructive electronic devices. These devices are based on natural phenomena, such as electricity and magnetism, and are well-known methods of geophysics. These methods are called ancient geophysical methods of locating antiquities.

The methods used for geophysical prospection are the *electrical* (2D or 3D images as in electrical resistivity tomography, ERT), the *magnetic* (with a dual magnetometer, a proton magnetometer, gradiometer), the *georadar* (or Ground Penetrating Radar, GPR), and aerial / satellite photography (Linford, 2006).

#### 4.1 GROUND PENETRATING RADAR (GPR)

GPR provides information on discontinuities in electrical properties of the ground using electromagnetic (EM) signals. The EM wave is transmitted to the ground by a contact with an antenna or transmitter. The EM wave passes through the ground at a speed determined by the electrical properties of the ground. When it undergoes a sudden change in the electrical properties of the subsoil it is reflected, refracted, and diffracted. A portion of the EM wave will return to the surface where it will be received from the antenna-receiver, displayed and recorded for processing in a subsequent step. The time elapsed between the transmission and reception of the EM signal refers to the distance between the discontinuity (target) and the surface where the signal is transmitted. This determines the depth of the buried target. This is achieved if the speed of propagation is known, which requires knowledge of the electrical properties of the sub-terrain. In a GPR surface scan, target detection and localization repeats with multiple traverses. In this way, an "image" is formed, as a kind of subterranean radiography not only with the detection and depth of the target, but also with their three-dimensional structure in the ground. (Conyers & Goodman, 1997).

#### 4.2 ELECTRICAL RESISTANCE PROSPECTION

Electrical prospection is based on the ability of matter to withstand the passage of electrical current through it. This resistance depends on the type of material. Thus, for each subsoil inhomogeneity, the electrical meter (potentiometer) gives a different resistance value (according to Ohm's law).

To carry out the electrical prospection of the ground it is necessary to channel a low frequency DC or AC through two metallic electrodes with low contact resistance. Its measurements difference of the dynamic field ( $\Delta V$ ) due to the flow of current from

different materials are made from two other electrodes. Ohm's law ( $I = V / R$ , where  $V$  is the potential,  $I$  is the current and  $R$  is its resistance allows the calculation of subsoil resistances. Considering one homogeneous and isotropic subsurface of infinite extent and taking into account geometrical coefficient of electrode arrangement ( $k$ ) one can determine the distribution of specific electrical resistance in the subsoil.

For non-homogeneous soils, where a deformation of the dynamic lines is observed of the electric field, the term 'apparent specific resistance' is used (eq.7).

$$\rho_a = k \cdot \frac{1}{\Delta V} \quad (7)$$

as a diagnostic indicator for examining its changes electrical resistance of the subsoil and enables the resistances of different materials to be compared in a standardized way.

Electric measurements can be made as: a) Vertical Electric Soundings - VES, b) Resistivity Profiling or Mapping, c) Electrical Resistivity Tomography - ERT.

Of the various devices used in electrical surveys (such as, Wenner, Square, Dipole-Dipole, Pole-Dipole, Twin, Schlumberger, etc.), the method of Dual Electrode Arrangement (Twin Probe) predominates in archaeological applications (Sarris et al., 2018, Ch.14).

#### 4.3 MAGNETIC PROSPECTION - MAGNETIC SUSCEPTIBILITY

An important parameter associated with the success of magnetic prospection is the magnetic contrast between architectural subterranean objects and the environment. Many times, human activity in a particular site results in an increase in the magnetic field soil properties. Iron oxides such as hematite, magnetite and maghemite are mainly responsible for the magnetic properties of soils of inorganic origin. Depending on their magnetic properties there are three main categories of materials: diamagnetic, paramagnetic and ferromagnetic. Every kind of material has unique magnetic properties, even those that we do not think of as being "magnetic". Different materials below the ground can cause local disturbances in the Earth's magnetic field that are detectable with sensitive magnetometers. The chief limitation of magnetometer survey is that subtle features of interest may be obscured by highly magnetic geologic or modern materials.

Magnetometers used in geophysical survey may use a single sensor to measure the total magnetic field strength or may use two (sometimes more) spatially separated sensors to measure the gradient of

the magnetic field (the difference between the sensors). In most archaeological applications the latter (gradiometer) configuration is preferred because it provides better resolution of small, near-surface phenomena. Magnetometers may also use a variety of different sensor types. Proton precession magnetometers have largely been superseded by faster and more sensitive fluxgate and cesium instruments.

Magnetic anomalies depend on the magnetization of the targets, their size and shape, and the depth at which they are. The magnetic surveys have been particularly successful in cases mapping of ancient settlements and cities or laboratory exploration installations, furnaces, ovens or installers, together with electrical techniques are systematically used in surface surveys for fast coverage areas and the correlation of surface ceramic sherds concentration with presence of subterranean monuments. Several types of magnetometer are used in terrestrial archaeology e.g. the proton precession magnetometers, the Fluxgate and cesium vapor (Aspinall *et al.*, 2008). Magnetic surveys are extremely useful in the excavation and exploration of underwater archaeological sites. The most common type of magnetometer used for marine surveying is the fluxgate magnetometer. Another common type is the newer proton precession magnetometer (Boyce *et al.*, 2004). Geophysical prospection has been also employed in non-conventional manners to tackle specific archaeological problems. Such problems might be, for example, the cases of locating tombs under tumuli embankments, assessing the moisture content in walls, the depth of fractures in sculptures, exploring the space behind walls, mapping the waterways along which the water drains out or in monuments, investigating in urban environment, etc. Relatively recently, the potential of this kind of operations has been the subject of numerous papers. However, many of these operations require a great amount of expertise and innovation (Tsokas *et al.*, 1994; Scollar *et al.*, 1990; Athanasiou *et al.*, 2007; Angelis *et al.*, 2018; Drahor *et al.*, 2015)

The non-evasive archaeo-geophysical surveys are most significant in unearthing buried antiquities and offer a cultural attraction and development. It contributes a great deal to sustainability. Hidden antiquities are revealed. Many projects have been implemented where geophysical investigations have helped to exhibit new archaeological sites, either underground or under the sea, and restrict illicit excavations and trafficking of antiquities, and protecting at-risk antiquities from either public works or environmental risks (Liritzis and Korka, 2019)

There exists a number of examples from all over the world, especially from countries with a rich cultural heritage. The numerous archaeo-geophysical

prospection (electrical, magnetic, georadar, remote sensing satellite imaging, and seismic sounding) first detect the buried target, which was followed by archaeological excavation, a study of the finds, and conservation and restoration tasks, to finally manage the opening the archaeological site and associated museum to the public, acquiring a sustainable character, such as in Italy with a significant project in South Etruria by the British School in Rome and Italian authorities<sup>4</sup> and in Egypt, China, Turkey and other parts of the world (El-Qady and Metwaly, 2019).

Reports of a number of examples exist from the Aegean region (Greece) that geophysical investigations have driven the archaeological investigations to a surgical kind of excavation, and at the same time contributed to the promotion of the sites and the general prominence of them (Sarris *et al.*, 2013).

The cemetery of the Roman Era of Europos, Northern Greece, comprises another example. It is situated at the foothill of a topographic table. In fact, the resistance prospecting at this particular area yielded distribution of resistances displayed as contours (Tsokas *et al.*, 1994).

Its interpretation is straightforward since pronounced high resistivity anomalies are surrounded by a rather uniform low resistivity environment. The "twin probe" electrode arrangement was used having the roving electrodes 1 m apart, one from the other, and 1 and 2 m, in line and cross line spacing, respectively. Therefore, it was a low-resolution survey aiming only to detect and map the position and the areal extent of large monumental tombs. Presumably, each one of the well-defined in space high resistance anomalies has been caused by such a concealed structure. On the other hand, the relatively sizeable blurred anomaly at the west side of the image was attributed to a hidden gravel deposition. In fact, after the excavations, the aforementioned interpretation proved true in all its predictions.

Last but not least, the variety and sophistication of data sources, sensors, and platforms employed in archaeological remote sensing have increased significantly over the past decade. Projects incorporating data from UAV surveys (drones, balloons), regional and research-driven Lidar surveys, the uptake of hyperspectral imaging, the launch of high-temporal revisit satellites, the advent of multi-sensor rigs for geophysical survey, and increased use of structure-from-motion (SfM) imply that more archaeologists

<sup>4</sup> <http://www.bsr.ac.uk/research/archaeology/completed-projects/tiber-valley-project/south-etruria-survey>) [ Geophysics Techniques. Available online: <http://www.bsr.ac.uk/research/archaeology/geophysics-2/geophysics-techniques> (accessed on 18 March 2019)

are engaging with remote sensing than ever (see, Fig.17). These technological advances continue to drive research in the specialist community and provide reasons for optimism about future applications, but many social and technical obstacles to the integration of remote sensing into archaeological research and heritage management remain. But in fieldwork for the past decade development and limitations, are ongoing, across three broad segments of landscape archaeology: data collection in the field, the current state of data access and archives, and processing and interpretation (Opitz et al 2018; Deiana et al., 2018; Scollar et al., 1990).

## 5. ARCHAEOASTRONOMY

Since prehistoric times, people have been paying close attention to starry skies and celestial phenomena, either for religious or worship purposes, or for using a calendar system used in agricultural pursuits and navigation. Many prehistoric and historical monuments appear to have been oriented towards the solar sunrise during the solstices and equinoxes or other very important festivals of the year.

Archaeoastronomy is the scientific study of the doctrines and practices concerning astronomy in ancient societies. It refers to the studies of monuments, ancient instruments (ancient technology), natural outcrops, natural or man-made structures, yet ancient literature, in relation to celestial bodies (e.g. the sky-scape that includes the sun, the moon, stars, constellations, meteoric phenomena). Proper celestial software determines the past skies at a particular place on Earth's surface. Archaeoastronomy thus offers novel data concerning the intangible heritage through the measurements of tangible heritage (Magli, 2009, 2013; Aveni 1989).

Thus, the study of how people in the past have understood the phenomena in the sky, how they used these phenomena and what role the sky played in their cultures, can be achieved via interdisciplinary and multiscientific methods. But the way to uncover evidence of past practices uses different subjects such as archaeology, anthropology, astronomy, mythology, statistics and probability, and history. Here perhaps the need to balance the social and scientific aspects of Archaeoastronomy is an extremely delicate matter, which, leads some (authors and journals) to unsounded, imaginable, uncontrolled speculation nurturing on lunacy (Ruggles, 1999). In the scientific orthodoxy Archaeoastronomy is based on measurements, sound processing and integrity, and any interpretation should be cautiously made but arbitraries strictly removed.

Orientation measurements made by a theodolite or compass and clinometer and GPS, or by remote tools (google maps), and employing specific soft-

ware for the determination of past skies, determine azimuths and declinations, following by error evaluation, statistical data elaboration, and celestial body positions, which in all, contribute in: a) the assessment of knowledge of observational astronomy by ancient people, b) obtaining information on religious (worship) beliefs and metaphysical perceptions, useful for examining their socio-political and religious structure, c) to decipher (interpret) ancient texts referring to celestial bodies or natural phenomena (Vlachos et al., 2018; Liritzis et al., 2017; Castro et al., 2015; Papamarinopoulos et al., 2014; Aveni, 1989;).

The following conclusions can be drawn from studies of astronomy in ancient cultures:

1) The ancient temples dedicated to the same deities are not necessarily oriented towards the same celestial goal. Rather, they relate to a celestial body interpretive corresponding to the particular attribute of the deity. For example, the god Apollo was not only the god of light, but was also attributed to him by other adjective designations that had to do with local festivals and attributes, such as hyperborean, Pythian, Smintheus, etc.

2) It is confirmed a wide choice of orientations in ancient temples; most temples are orientated mainly in relation to the sun, while zodiac signs, astronomical cycles are evident in various parts of the world

3) The orientations in the direction of solar stands (solstices, equinoxes), lunar standstills, and star rise / set, provide a wider variation of azimuths along the solar horizon, possibly reflecting the existence of a complex calendar of particular importance on certain days of the year.

4) A possible connection between religious and / or mythological elements with specific celestial bodies can be identified. Such an accumulative knowledge of contemplating astrology became observational astronomy, which was initially used for practical purposes in their daily lives, but also for ritual / sacred purposes, that progressively ended up being a rational process, that is, science.

5) The particular interest of modern archaeology is not so much the monuments themselves, but the people. Archaeologists are no longer solely focused on archaeological materials but are trying to understand ancient and especially prehistoric society by studying the wider activities of people reflecting on the presence of settlements, the distribution of artifacts, along with environmental data, their spatial distribution as an indication of human activities in relation to the landscape and its change over time. Archaeoastronomy or astronomy of cultures contributes precisely to this point, to answer questions about what ancient societies believed and thought, through the exploration of monuments by astronom-

ical methods, hence contributing to the intangible heritage.

6) Notwithstanding any scientists' doubts as to whether the laying of foundations in the construction of megalithic monuments and temples is intentional or random, we would note the following: (a) the ever growing witness in fieldwork measurements indicates that the orientation was not accidental, (b) it is an utopia the extreme precision in orientation measurements made by specialists (in seconds or minutes of a degree!) which may overestimate the knowledge of prehistoric craftsmen, (c) sophisticated Archaeoastronomy occasionally impose onto archaeologists and non-experts, and consequently archaeologists-users of the result, a submissive disposition, in front of their authority, lead to wrong impressions in their interpretative stage (Castro *et al.*, 2015; Liritzis *et al.*, 2017; Liritzis & Vassiliou, 2003)

## 6. BIOMOLECULAR ARCHAEOLOGY

### 6.1 PALAEOGENOMICS AND ARCHAEOLOGY

With the decoding of the human reference genome and the advances in genotyping and sequencing (next-generation sequencing, NGS), the ancient DNA research has benefited and reached unprecedented details on the study of past populations. This exponentially evolving field is rapidly affecting diverse scientific fields besides biotechnology and biomedical studies. In parallel, isotopic studies can reconstruct mobility and palaeodiet. Under this light palaeogenetics and a series of analytical techniques such as isotopic studies have offered significant data and fresh ideas on long-lasting archaeological debates (Evison, 2014).

Ancient DNA studies started in 1984 when the first recovery (221 bp) of mitochondrial DNA from an extinct member of the horse family (quagga) took place (Higuchi *et al.*, 1984). The term "ancient" DNA refers to the extraction of a small amount of genetic material from extinct and extant humans, animals and plants. When an organism dies, DNA begins to degrade. However, under certain environmental circumstances, such as dehydration, low temperature and high salt concentration, agents that damage DNA may be incapacitated and DNA can be preserved for some thousand years (Hofreiter *et al.*, 2014). However, even when DNA does survive, it will be damaged, broken in small fragments and chemically modified. Due to the high amount of damage to ancient DNA and the relatively small recovered amount, the extraction process and the study of prehistoric organisms was a difficult and laborious task. Moreover, the poor preservation did not allow, the analysis of large parts of nuclear DNA

but rather of targeted parts of only mtDNA. Mitochondrial DNA appears in more copies in each cell compared to nuclear DNA that comes in only one copy per cell. All these factors limited the possibility of profound inferences, and therefore aDNA studies remained for many years an important but rather exotic research field.

Within the last ten years, due to the advancement of sequencing technology (NGS), ancient DNA analysis has evolved remarkably and could demonstrate the full potential of the method. The problems of sample contamination, the low endogenous DNA content and the prohibitive costs have been largely overcome. In 2010, the nuclear genome of the Neanderthals (Green *et al.* 2010) and the draft genome of a 4,500 thousand years old Eskimo (Rasmussen *et al.* 2010) were published. At the same time, genomes of past and present domesticated and extinct animals such as mammoths, cave bears, marsupial wolves have been produced recreating the palaeoenvironment of the planet. The time limit of DNA recovery has been pushed back. In permafrost, researchers could isolate DNA from a Pleistocene horse dated to approximately 560.000-780.000 years before present (Orlando *et al.*, 2013).

One of the major questions that aDNA research tries to answer is the biological affinities of past population groups. From a biological and evolutionary perspective biological affinities triggered by admixture, migration and mobility of people have shaped the human past and thus their understanding are decisive on making past inferences. Biological affinities can be studied from a population genetic perspective on inter-continental or continental level or on narrow local level such as kinship and mating. Today there are many examples of palaeogenomic studies about the migratory movements of past humans (Prüfer *et al.*, 2014; Raghavan *et al.*, 2014a; Raghavan *et al.*, 2014b; Rasmussen *et al.*, 2014; Raghavan *et al.*, 2015; Posth *et al.*, 2016; Unterländer *et al.*, 2017). One example is the genetic basis of today's European population on the advent of the Neolithic. The "Neolithic agricultural revolution or Neolithic demographic transition" and the role of the hunter-gatherers and the first farmers has been an active scientific debate by historians, anthropologists, archaeologists, biologists and geneticists for more than a century. This process, which describes the transition from hunting-gathering to agriculture and permanent settlement, have had a decisive influence on the evolution of human history and has changed the demographic and cultural profile of the human population.

Based on mtDNA data and low coverage genomic data it was shown that local hunter-gatherers were diverse from the first farmers in central Europe and

that there was genetic affinity among farmers in central European and modern populations of western Eurasia (Bramanti et al. 2009; Haak et al., 2005; Haak et al. 2010). There was also genetic discontinuity between hunter-gatherers and early farmers in Scandinavia and greater genetic similarity of the Neolithic farmers with modern European populations of the Mediterranean (Skoglund et al., 2012; Malmström 2009). In western Europe, genetic analyses in skeletal material of the late Neolithic of the Iberian Peninsula indicate a migratory wave of male population, mainly through the Mediterranean route (Lacan et al. 2011) and differences with the first farmers in central Europe (Hervella et al. 2012).

A combination of genetic and isotopic data from the archaeological site of Blätterhöhle in central Germany with Mesolithic and Neolithic skeletal material showed that the descendants of the local Mesolithic hunter-gatherers maintained for 2000 years after the introduction of the Neolithic way of life into their area, the same foraging subsistence strategy, without adopting the Neolithic innovations (Bollongino et al. 2013). In contrast, farmers who lived next to the Mesolithic groups and exploited the same geographic area, followed a standard diet eating domesticated animals and plants. Although generally was considered that hunter-gatherers became extinct after the arrival of agriculture, it is proven that they lived alongside with farmers for a very long time. While this model certainly does not apply to the whole of Europe, it offers an interesting insight into the many different models of neolithization process that exist in Europe.

The first whole genomes were published in 2014 from a 7,000-year-old farmer from Germany and eight, 8,000-year-old hunter-gatherers from Luxembourg and Sweden (Lazaridis et al. 2014). The study showed that present-day Europeans derive from at least three highly differentiated populations: west European hunter-gatherers, who contributed ancestry to all Europeans but not to Near Easterners; ancient north Eurasians related to Upper Palaeolithic Siberians, who contributed to both Europeans and Near Easterners; and early European farmers, who were mainly of Near Eastern origin but also had west European hunter-gatherer related ancestry (Lazaridis et al., 2014; Jones et al., 2015). Since then, important studies from the core of the Neolithic expansion took place where some of the earliest evidence for farming is found. Analysis on the first Neolithic farmers from northern Greece (prehistoric sites of Paliambela, Revenia and Kleitos) and north-western Turkey (prehistoric site of Barcin) demonstrated a direct genetic link between Mediterranean and Central European early farmers (Hofmanova et al. 2016). The study of the Neolithic Aegeans has also

shed light on the migratory routes of the Early Neolithic farmers suggesting two independent colonisation routes, one through the Balkan peninsula and one through the Mediterranean Sea. The discussion on the Neolithic processes was enriched by the genomic data from animal and plant domestication and the effects of those cultural process in human biology (lactase persistence, celiac disease) and cultural practices.

The genomic data produced from prehistoric individuals represent thousands of loci, under various selection pressures, and reflect the complex and gradual nature of evolution. Increasing number of studies reveal multiple events of admixture and gene flow. Today is possible with the analysis of individual genomes, to make inferences on more archaeologically oriented research hypotheses, such as mating system, social practices, group size, inbreeding rate, degree of polygamy or endogamy. Runs of homozygosity (ROH), for example, are long sections of an individual chromosome, which are homozygous (identical) both on their maternal and paternal copy. Depending on their length and their number in an individual's genome, we can interpret the degree of consanguinity of the individual's parents or the size of the individual's community. Genetic diversity of specific maternal (mitochondrial DNA) or paternal (Y-chromosomes) lineages combined with genomic data can shed light on the family and mating strategies (patrilocal vs. matriloc). Basic family structures and kinship patterns can be reconstructed and shed light on the broader social organization of ancient communities.

Similarly, the study of disease in the past can be now fully explored. Pathogenic bacteria enter the bones and teeth, leaving traces of their DNA in the skeleton. By extracting and sequencing the bacterial and viral DNA, it is possible to confirm the presence of a disease, e.g. *Mycobacterium tuberculosis*, *Yersinia pestis* (Bos et al., 2014; Devault et al., 2014; Thalmann et al 2013). Sequencing of the bacterial DNA can help us understand the origin and evolution of the disease and their changes over time. This data can be interrelated to literary sources in order to reconstruct the history of major epidemics afflicting mankind from antiquity to the present, e.g. the plague of ancient Athens or the Black Death epidemics (Andam et al., 2016).

Palaeopathological genomic analysis is investigating the co-evolution of humans and pathogens. By sequencing candidate genes, exomes or whole genomes directly from ancient humans and pathogens, it is feasible to directly observe the genetic modifications and adaptations of human populations exposed to pathogens and epidemics, and associate these with specific pathogen mutations or variants

through time and space (Hofreiter *et al.*, 2014). A good example is the analysis on calcified dental plaque (dental calculus) preserved for millennia on human teeth that enables the simultaneous investigation of pathogen activity, host immunity, and diet. Dental calculus entraps biomolecules from all domains of life and viruses and the analysis on ancient oral microbiome sheds light on the intimate evolutionary relationship between humans and their microbes (Warriner *et al.*, 2013).

## 6.2 ISOTOPIC ANALYSIS

Isotopic studies consist a globally growing field of research because of the vast range of topics they cover – only a few of which are being discussed in the following paragraphs. The isotopic approach to human past has been used by archaeologists since the 1970-80s, initially for palaeodietary reconstruction (DeNiro and Epstein 1978, 1981, Chisholm *et al.*, 1982), and later on (e.g. Ericson, 1985), for purposes of tracing ancient mobility routes. Because of its importance and the broad scientific scope it covers, stable isotope analysis has become today a sub-discipline of its own (Bogaard and Outram, 2013).

Archaeologists traditionally use isotopes for diet and mobility investigations. Chemical variation in hard tissue depicts variation in diet, nutrition and disease, but also in environmental factors, revealing places of residence and migration patterns (Katzenberg, 2008). Contemporary research on stable isotopes has also started to employ sophisticated statistical analyses. Froehle *et al.*, (2012) created a multivariate model for reconstructing human diet, expanding on the earlier bivariate carbon model by adding bone collagen nitrogen values to the analysis. Fernandes *et al.*, (2015) has used a Bayesian model to predict with more accuracy the source of dietary protein in individual consumers.

By the term isotopes we infer to alternative forms of certain chemical elements that have similar numbers of protons and electrons in their nuclei, but differ in their number of neutrons. Stable isotopes, unlike unstable or radioactive ones, do not decay over time. Isotopic analysis includes the method of bulk isotopic analysis of carbon (C), nitrogen (N), Sulphur (S) and the newly explored Zn and incremental analysis of dentine. The analysis of stable carbon isotope ratios ( $\delta^{13}\text{C}$ ) provides information about plant consumption, nitrogen ( $\delta^{15}\text{N}$ ) reflects the intake of animal protein. The analyses of stable sulphur ( $\delta^{34}\text{S}$ ) as a multi-isotopic approach provide information about the intake of marine and freshwater food whereas Zn is newly explored as a proxy for animal protein consumption (Jaouen *et al.*, 2018). Incremental analysis of dentine in which consecutive slices of tooth dentine are measured for C and N iso-

topic values reveals variations in childhood diets (Beaumont and Montgomery, 2016) as well as breast-feeding and weaning practices. In contrast to bone, primary dentine does not remodel and can provide information on radical, short term changes in diet that can then be used to answer more detailed questions. The method records specific periods of food shortage, and can therefore be linked - chronologically or causatively- to historical events mentioned in written records.

Strontium (Sr) isotope ratios found in human teeth and bone directly reflect the composition of consumed water, plants and animals, which in turn reflect the bedrock composition of a geographic region (e.g. Ericson, 1985; Slovak & Paytan, 2012). By taking into account geo-specific isotopic ranges, it is possible to assess human mobility (e.g. Bentley *et al.*, 2004, 2006, 2007, Budd *et al.*, 2004; Buzon *et al.*, 2007). Analyses on Sr isotope ratios have been performed on selected, archaeologically indicated cases. Strontium ratios can trace mobility with generation-specific precision. At the same time, due to the fact that Sulphur isotopic composition is also determined by underlying local bedrock and atmospheric depositions, S isotopes can also be used in mobility studies. Therefore, a correlation of C, N, S and Sr can assist in tracing individuals by detecting whether the data reflect a true marine diet or rather an outlier due to migration (Vika, 2009). A sound understanding of the geological formations together with the bioavailability of strontium at each site need to be fully reconstructed, before anyone can be effectively identified as non-local. At the same time, strontium will rarely identify a single origin of a non-local individual which may restrict the method under certain conditions.

Carbon isotopes, also, enter the food chain as herbivores consume plants, and oxygen isotopes enter the food chain from consuming meteoric water and water from the diet. Examining the  $^{12}\text{C}/^{13}\text{C}$  isotope ratio, it is possible to determine whether animals ate predominantly C3 or C4 plants. Potential C3 food sources include rice, tubers, fruits, nuts and many vegetables, while C4 food sources include millet and sugar cane. This process ends with the organism's death, from this point on isotopes no longer accumulate in the body but do undergo degradation. For best result the researcher would need to know the original levels, or an estimation thereof, of isotopes in the organism at the time of its death.

Atomic C:N ratios were calculated according to the following equation: Atomic C-to-N =  $(14/12) \times$  (Weight percent C:N). The atomic C:N ratios of human bone collagen samples is expected to fall within the modern collagen range of 2.8 – 3.6. Human dietary composition is addressed employing standard

bone collagen  $\delta^{13}\text{C}$  values of  $-23\text{‰}$  for a 100 percent C3-based terrestrial diet,  $-13\text{‰}$  for a 100 percent marine-based diet, and  $-7\text{‰}$  for a 100 percent C4-based terrestrial diet (van der Merwe et al. 1988, Pate and Schoeninger 1993). The validity of obtained values from archaeological sites must be confirmed using archaeological faunal controls derived from the site and closely adjacent area. Because past atmospheric  $\text{CO}_2$  was approximately  $2\text{‰}$  more positive than that in the modern atmosphere (Peng and Freyer 1986, Bada et al., 1990, Marino and McElroy 1991), a  $2\text{‰}$  correction factor is employed in relation to the use of modern faunal controls. (see example for Pompeii, Italy, in Pate et al., 2016).

Regarding isotopic effects of carnivory and herbivory on protein of chicken and egg carbon it has been examined the different diets of  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  and carbonate  $\delta^{13}\text{C}$  measured on egg and bone from

hens. Herbivorous hens had a  $+14.3\text{‰}$  spacing between egg albumen and shell  $\delta^{13}\text{C}$ , compared to  $+12.4\text{‰}$  for omnivorous hens, and  $+11.5\text{‰}$  for carnivorous hens. The bioapatite-collagen  $\Delta^{13}\text{C}$  spacing was measured as  $+6.2\text{‰}$  for herbivorous hens, and calculated as  $+4.3\text{‰}$  for omnivorous hens, and  $+3.4\text{‰}$  for carnivorous hens - similar to observed mammalian herbivore and carnivore bioapatite - collagen  $\Delta^{13}\text{C}$  differences. It is concluded that a shift in diet composition from herbivory to carnivory in a single species does alter the bioapatite - collagen carbon isotopic spacing. Data strongly suggest that this results from differences in the  $\Delta^{13}\text{C}_{\text{bioapatite-diet}}$  spacing, and not that of  $\Delta^{13}\text{C}_{\text{collagen-diet}}$ . (O'Connell and Hedges, 2016). Ecologists have long used the  $\delta^{15}\text{N}$  value as a measure of an animal's position in the food web, or "trophic level" (Fig.12).

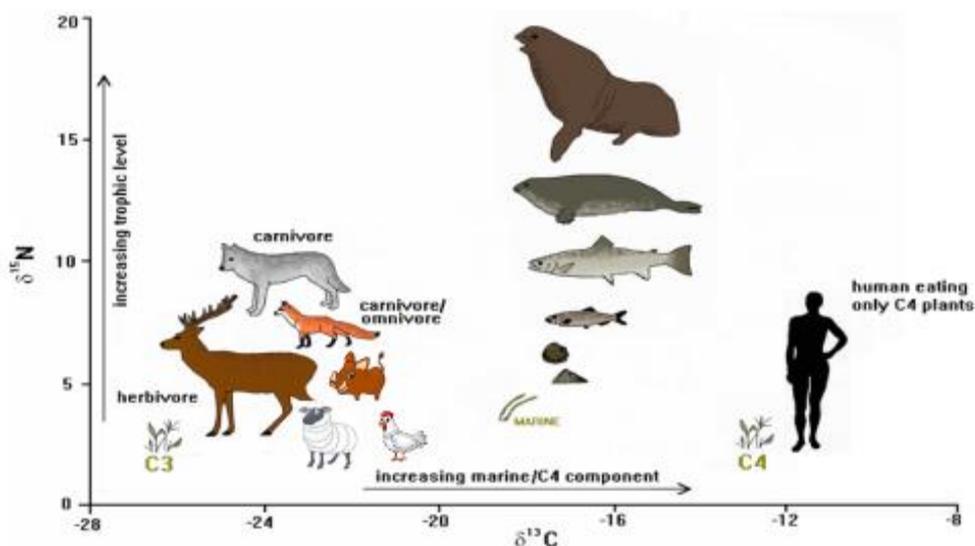


Figure 12. Simplified summary of stable carbon and nitrogen values for terrestrial and marine ecosystems (values are for flesh; to convert to bone collagen values,  $5\text{‰}$  should be added to  $\delta^{13}\text{C}$  values;  $\delta^{15}\text{N}$  values are the same for flesh and bone) (see: adapted from: Dr Svetlana Svyatko, Information about Stable Carbon and Nitrogen Isotope Analysis; <http://www.chrono.qub.ac.uk/IRMS/>; Schulting 1998).

Interesting is the presence of poultry (chicken, hens) in archaeological excavations with measured variation spanning for  $\delta^{13}\text{C}$   $-19\text{‰}$  to  $-20\text{‰}$  and  $\delta^{15}\text{N}$   $2-4\text{‰}$  (for egg, tissue, muscle, meat) (Skipityte et al., 2016; Denadai et al., 2019) (see, Fig.12).

Isotopic measurements including isolation from the preserved bone of collagen (C and N) and bone apatite (C), and chemical elements as strontium, barium and macroelements, like Ca and P, are made. Analyses of Ba, Sr and Ba/Sr, Ba/Ca and Sr/Ca ratios are used for reconstructing the weaning process and for determining the difference between marine and land diet. However, chemical signals and reconstruction of life strategies from ancient human bones

and teeth is not an easy task (Szostek, 2009; Bocherens and Drucker, 2003).

In palaeodiet investigations the question is where the food comes from and the isotopic imprints that the ecological processes involved in food production leave on those foods. The atoms of each of the elements that make up our food move through an elemental cycle from inorganic to organic forms, and back again. The chemical and biological reactions inherent in each elemental cycle have the potential for isotopic fractionation and cause a natural partitioning of isotopes through the environment. Although elemental cycling is far outside of the purview of most nutritionists, it is always instructive to

consider where our food comes from, and understanding how and why isotopes vary in our foods is important to understanding the strengths and the limitations of stable isotope ratios as biomarkers of diet (O'Brien, 2016).

To obtain an accurate picture of palaeodiet it is important to understand processes of diagenesis that may affect the original isotopic signal. It is also important for the researcher to know the variations of isotopes within individuals, between individuals, and over time (Kontopoulos *et al.*, 2019).

Socio-economic factors, as well as differences in food access between sexes and age groups may be distinguished through palaeodietary analysis. In the same way dietary habits may reveal relations between biology, culture, and socio-economic status of individuals in the community (Papathanasiou & Fox, 2015). For the above reasons isotopic studies for palaeodietary reconstructions have been extremely exploited by archaeologists and anthropologists worldwide.

Last, oxygen isotopes in human bones and teeth provide useful information regarding the sort of collective life history of the deceased individuals (e.g. place of origin of an individual during the childhood and when adult). Isotopic data including

$\delta^{18}\text{O}$  and Sr indicated that the Iceman (Otzi) spent his entire life in the area south of the discovery site in the Alps making occasional trips to the northern alps region as hunter-gatherer (Muller *et al.*, 2003).

The mineral phase of dentine, enamel, bone in the phosphate or carbonate fractions, offer useful fingerprints of oxygen isotopic signatures (Kohn and Cerling, 2002). For both fractions of phosphate and carbonate, the  $\delta^{18}\text{O}$  values are related to those of environmental water through drinking water. However, there are complications due to the incorporation of water from food, and mixture with oxygen from respiration. This leads to varying species-dependent relationships between  $\delta^{18}\text{O}$  values in carbonate or phosphate and  $\delta^{18}\text{O}$  values in environmental water taken by the organisms. However, both  $\delta^{18}\text{O}$  values are linked by a clear relationship independent of the organism. Variation in oxygen isotope ratios studied from terrestrial environments is ascribed to environmental temperature changes, with warmer weather resulting in more positive  $\delta^{18}\text{O}$  values and cooler weather resulting in more negative  $\delta^{18}\text{O}$  values in meteoric waters (Bocherens and Drucker, 2013; Evans *et al.*, 2012; Price *et al.*, 2012). (see, Fig.13).

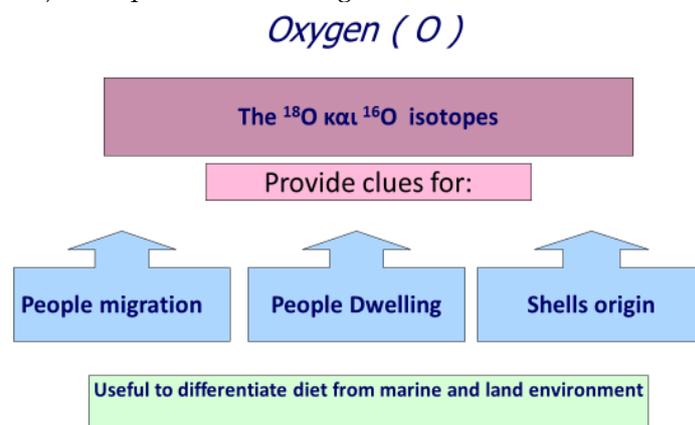


Figure 13. The useful information obtained from stable oxygen isotope for humans and marine shells (Liritzis & Vafiadou ©)

## 7. CONSERVATION SCIENCES

Several times, curators/conservators are in front of an artifact, monument, or an archaeological/historical evidence of cultural heritage that must be repaired, conserved or restored. Major questions rising are from what materials are made, what techniques were used, what degradation processes have been developed and what products should be selected for treatment. If the applied materials and techniques are known, the conservation is safer due to the selections of the proper materials and methodology.

Modern conservation science has been developed following, in general, two main streams (Giorgi *et al.*, 2010): (i) the development of methods for the identification of materials in objects of cultural and artistic value and the investigation and monitoring of their degradation processes, developed because of ageing. (ii) The research for new scientific methods and materials for effective and sustainable interventive and preventive conservation strategies. As described above, the two streams are apparently interconnected as before the application of a conservation treatment, it is important to know the materials of the object and its degradation processes. Conse-

quently, conservation science is an interdisciplinary field where physics, chemistry, biology and materials science meet, along with their multiple sub-fields in engineering, nanosciences and nanotechnology (Creagh and Bradley 2000; Stuart, 2007; Baglioni and Chelazzi, 2013; Hosseini and Karapanagiotis, 2018).

The characterization of the original materials and their degradation products, which are found in objects of the cultural heritage (stream i), has been benefited by the remarkable progress of the physico-chemical techniques. In late years/decades non-destructive techniques gain interest in compositional analysis of such artifacts.

The first approach for studying an object is through visual observation. This obviously includes a very good photograph, but besides that, close up and macro photos are very useful because specific areas of particular aesthetic or artistic interest are being isolated giving a different perspective in details, with the possibility of using not only reflected light, moreover raking or grazing light (Kirsh and Levenson, 2002) and all that in visible radiation, as well as in invisible, such as the highly penetrated infrared or the ultraviolet for surface detection.

The invention of Electron Microscopy in the first half of the 20th century has offered in the last decades powerful and easy-to-use instruments, such as Scanning, Transmission and Field Emission Electron Microscopes which are extensively used for imaging samples from objects of cultural and artistic value (Creagh and Bradley, 2000). The application of Scanning Probe Microscopy in conservation science is rather limited. On the contrary, Optical Microscopy and its multiple versions (e.g. stereomicroscopy, fluorescence microscopy) is a major tool to study all kinds of objects and/or samples and to carry out various studies such as, for instance, the investigation of cross sections of multi-layer pictorial works of art (Karapanagiotis et al., 2013). Other imaging techniques, which have been used in conservation science for several decades to reveal object's state of preservation, are UV Reflectography, IR Reflectography and X-radiography. More recently, the non-invasive identification of Egyptian blue was achieved by Photo-induced Luminescence (Verri, 2009), whereas the layers of paintings can be revealed via non-sampling procedures using tomography, such as Optical Coherence Tomography (OCT).

The interaction/emission phenomena of X-rays with/by the matter at the atomic scale have offered a rich variety of techniques which are useful to identify mainly inorganic materials and to study their degradation mechanisms. Besides X-radiography, other X-ray techniques which are widely used in conservation science are similar to those archaeometry uses, in general, i.e. X-ray Fluorescence Spectroscopy

(XRF), Scanning Electron Microscopy coupled to Energy Dispersive X-rays (SEM-EDX), X-ray Photoelectron Spectroscopy (XPS), Proton Induced X-ray Emission (PIXE) and X-ray diffraction (XRD). Some of these instruments are portable and can be applied in a non-sampling fashion, implying that they have no impact on the integrity of the studied object. The most prominent example is the portable XRF which has become a major tool to study manuscripts, books and other objects for which the removal of even tiny samples is practically prohibited.

Other ways of using X-rays began to be displayed for the in-depth imaging of painted surface objects, such as strati-radiography (Van Asperen de Boer, 1976), Electron emission, K-edge dichromography (Baldelli et al., 2006) or the very important stereo-X-ray (Van Asperen de Boer, 1976) extremely useful for paintings painted on both sides. Great effort has been made over the last decades for the expansion of ways to visualize underlayer painted layers, like the Synchrotron Radiation Based XRF and macro-XRF and Confocal 3D Micro-XRF (Bratitsi et al., 2019).

Atomic spectroscopy is another set of techniques which is useful to study inorganic materials and, more important, to carry out quantitative elemental analyses. The latter can sometimes enable to determine the provenance of the studied objects e.g. ceramics. Atomic Absorption Spectroscopy (AAS), Atomic Emission Spectroscopy (AES) and the recently emerged and more sensitive, Laser-induced Breakdown Spectroscopy (LIBS), have offered important results in the study of the tangible cultural heritage.

Holographic interferometry is a promising diagnostic tool (Bonarou A. Et. al., 2001) for the analysis of the condition of historical structures (Kosma et al, 2018) since we can detect small displacements and deformations.

In the rich world of molecular spectroscopy there are two techniques which have been most commonly used in conservation science: Fourier Transform – Infrared Spectroscopy (FTIR) and Raman Spectroscopy (see above). The latter has become the standard technique to identify inorganic colourants (pigments) in works of art (Bell et al., 1997; Vandenaabee, 2012). A major limitation is that Raman Spectroscopy is often plagued by a strong fluorescent background which, in principle, can be reduced by employing Surface Enhanced Raman Spectroscopy (SERS).

FTIR spectroscopy is a very popular technique which is extensively used to identify natural inorganic and organic materials (e.g. binders, adhesives, varnishes etc.) and to characterise synthetic materials used in conservation practice (Derrick et al., 1999). It

is based on the ability of compounds to absorb infrared light (IR) (Yan *et al.*, 2019).

It began with Vidicon analogue tubes with high penetration depth and evolved to InGaAs (sensitive to radiation between 900 and 1700 nm), but quite recently Multispectral and Hyperspectral imaging has appeared offering a number of views recorded at different wavelengths (Bratitsi *et al.*, 2019).

Other non-sampling molecular spectroscopic techniques employed frequently to study cultural heritage objects, are Fibre Optics Reflectance Spectroscopy (FORS) and Fibre Optics Fluorescence Spectroscopy (FOFS).

Mass Spectrometry (MS) operating with various (i) ionization sources, such as for instance Electron Ionization (EI), Electrospray Ionization (ESI) and Matrix-assisted Laser Desorption/Ionization (MALDI) and (ii) mass analysers, such as for instance, quadrupole, ion trap and time of flight, is extremely powerful in the analysis and identification of natural organic materials including oils, proteins, gums, waxes and natural resins. Pyrolysis-Mass Spectrometry (Py-MS) is another widely used MS instrumentation which can detect a wide range of different compounds with a single analysis. For the investigation of inorganic materials Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) has been frequently employed whereas Secondary Ion Mass Spectrometry (SIMS) can provide compositional results for inorganic and organic species. Mass Spectrometry is a sampling invasive method. However, it can provide quantitative results which are sometimes important, particularly in the analysis of organic materials.

The analytical capabilities of MS can be extended when it is connected with chromatographic techniques such as liquid chromatography (LC) and gas chromatography (GC) (Colombini and Modugno, 2009). LC-MS is used mainly for the identification of natural organic colourants, that are dyes in textiles and lakes in paintings, whereas GC-MS is used for the analysis of various organic materials such as binders and varnishes. LC can be also accompanied by a Photo-Diode-Array (PDA) detector providing LC-PDA systems which are very effective in the detection of organic colourants in objects of the cultural heritage (Hofenk- de Graaf, 2004).

Regarding the research activities for the development of new scientific methods and materials for effective and sustainable conservation strategies (stream ii), the emerging nanotechnologies opened new avenues for the production of materials with improved properties. Some examples of the advanced materials which were recently produced are the following. Micellar solutions, microemulsions and easily "peelable" gels have been produced and used successfully as cleaning agents for pictorial ob-

jects (Baglioni and Chelazzi, 2013). Green methods for textile cleaning and disinfection using supercritical fluids have been developed at a laboratory scale (Aslanidou *et al.*, 2004). Biomimetics, superhydrophobic and water repellent coatings and improved, nanocomposite consolidants offered new perspectives for the protection of natural stone (Hosseini and Karapanagiotis, 2018).

Nanoparticles offered new materials for paper deacidification (Baglioni and Chelazzi, 2013) and painting conservation (Salama *et al.*, 2018). Photocatalytic nanomaterials (e.g. TiO<sub>2</sub>) were added in products of conservation science to induce self-cleaning properties. Likewise, new antimicrobial and anti-graffiti materials have been emerged for the protection and preservation of the cultural heritage (Hosseini and Karapanagiotis, 2018), and improved materials such as polysiloxane in conservation and protection of stone monuments and other outdoor objects of the cultural heritage enhancing the inherent hydrophobic character (Manoudis *et al.*, 2017).

The use of laser technology has given immense possibilities to the conservation science, by cleaning archaeological materials (Kennedy *et al.*, 2004; Siano *et al.*, 2005) removing aged varnishes (Pouli *et al.*, 2008) and from paintings and past conservation treatments (Pouli *et al.*, 2009) with high control and accuracy (Kautek *et al.*, 2003), as well as, applying analysis and diagnosis in many artworks of complex structure.

Major applications for this field are on inorganic (stone monuments, ceramics, glass, wall paintings) and organic origin (paper, textile, parchments, bones) to mention but a few.

Some issues include pigment analysis, authentication, corrosion.

Pigment and ink analysis. Is focused on the identification of the pigments and inks used in art works or manuscripts. Is based on the assumption that every color has a unique chemical formula and therefore a unique spectrum - a fingerprint - when an analytical technique is applied. For example, the manuscript D.I.21 kept at Biblioteca Nazionale Universitaria in Torino, better known as Messale Rosselli, is one of the richest fully illustrated missals surviving from the mid-14th century. The full set of colourants was identified, highlighting the systematic use of precious pigments such as lapis lazuli, cinnabar and gold, a feature reinforcing the symbolic value of the manuscript; in addition, less valuable but interesting dyes such as brazilwood and folium were also identified, used either pure or in a mixture with pigments in order to obtain a wide range of hues (Cala *et al.*, 2019).

Authentication. A lesser known application of archaeometry in conservation science and pig-

ments/ochres analysis is the possibility of authentication of art works. This method is based on the assumption that a painter uses constantly the same pallet of colorants and pigments. The chemical composition of the color pallet is well identified by non-destructive analysis and the "footprint" of his methods are established. Therefore, in an unsigned painting the reconstruction of the color pallet should reveal the artist. For example, recently, 21 oil paintings created in the nineteenth century by Polish painter and academic professor Rafał Hadziewicz (1803–1886) were investigated by various spectroscopic techniques (XRF, SEM, FTIR and more). The recognition of the artist's palette and the pigments used was significant due to Hadziewicz's influence on subsequent generations of Polish painters. The artist's palette chromophores (i.e. ultramarine, cobalt and Prussian blue, goethite, hematite, and magnetite from natural ochres, lead basic carbonate, lead carbonate, anatase, zinc carbonate hydroxide, zinc oxide, lead chromate, carbon, cadmium sulfide, copper acetate arsenite, verditer, and more) were identified mainly by Raman spectroscopy. The obtained results can provide support in authentication studies (Doleżyńska-Sewerniak & Klisińska-Kopacz 2019). Characterization by Raman spectroscopy of 20th century art materials from the Lasar Segall Museum, Brazil, was studied including collection of art materials, painted works and techniques, using materials and documents of the early 20th century. All the color palette found in paintings from 1919 is closely related to the characterized pigments from the museum, but one, the violets identified as organic not natural mineral. The results assist future conservation, dating, and authentication procedures of the works (Mendes et al., 2019).

The analysis of pigments and inks, can classify a work of art into the period which it belongs, while enabling it to prove that a work is fake even if it is signed, such as a high-value painting attributed to the famous American painter Jackson Pollock, which eventually proved to be fake, because Raman analysis showed that it contained a red color that did not exist on the market at the time the painting was supposed to have been crafted. Also three other paintings attributed to the same artist were examined with Raman spectroscopy, SEM-EDX, LDI-MS, FTIR, py-GCMS and C-14 dating and has been proved that they contained pigments and media available only after Pollock's death (Khandekar et al., 2010).

The evolution of technology prevents counterfeiters from deceiving prospective buyers, as scientists are now able to determine with absolute certainty whether a painting has been repainted and what elements have been added since its original creation and of course if the artist's signature is fake.

**Corrosion identification.** Several cultural heritage materials, mainly monuments, are made from large parts of marble and/or metal alloys and are placed in central spots in cities. These monuments need a continues conservation and monitoring their condition. In the early stages of conservation conservators need to know the exact chemical composition of the corrosion results in order to select the proper curing methodology (Aucouturier and Darque-Ceretti, 2007).

## 8. GEOARCHAEOLOGY

The geoarchaeology or archaeological geology is a rapidly growing interdisciplinary field in which archaeologists and geologists attempt to use the tenets of both their sciences to solve problems of mutual interest. This involves the application of the principles of geomorphology, sedimentation, isotope geochemistry and petrology. As Hertz & Garrison (1998) stressed, it is less what one calls the use of earth science techniques in archaeology than what one realizes from their application. The practice of modern archaeology demands an understanding of earth science. Only by this way archaeology integrates to the wider field of natural sciences in lieu of being a mere branch of art and humanities.

The term first introduced in the beginning of 70s, it has already a significant past and relates to other disciplines as the geology, geography, environment, archaeology. Karl Butzer (1971) first conceived the term in the dual interdisciplinary field of environment and archaeology. First monograph of geoarchaeology was by Davidson & Shackley (1976) and later by Rapp and Gillford (1986).

In geoarchaeology an essential issue is to understand the meaning of proxy data. (<https://www.ncdc.noaa.gov/news/what-are-proxy-data>). Proxy data are preserved physical characteristics of the environment that can stand in for direct measurements. In science, it is sometimes necessary to study a variable which cannot be measured directly. This can be done by "proxy methods", in which a variable which correlates with the variable of interest is measured, and then used to infer the value of the variable of interest.

A prerequisite task, however, is the definition of terms, the meaning of the paleoenvironment, giving examples of using proxy data, the palaeoenvironmental reconstruction in local and regional scale, and the palaeoclimatic zones in different regions of the world (Fig.14).

In paleoclimatology, or the study of past climates, scientists use what is known as proxy data to reconstruct past climate conditions. These proxy data are preserved physical characteristics of the environment that can stand in for direct measurements.

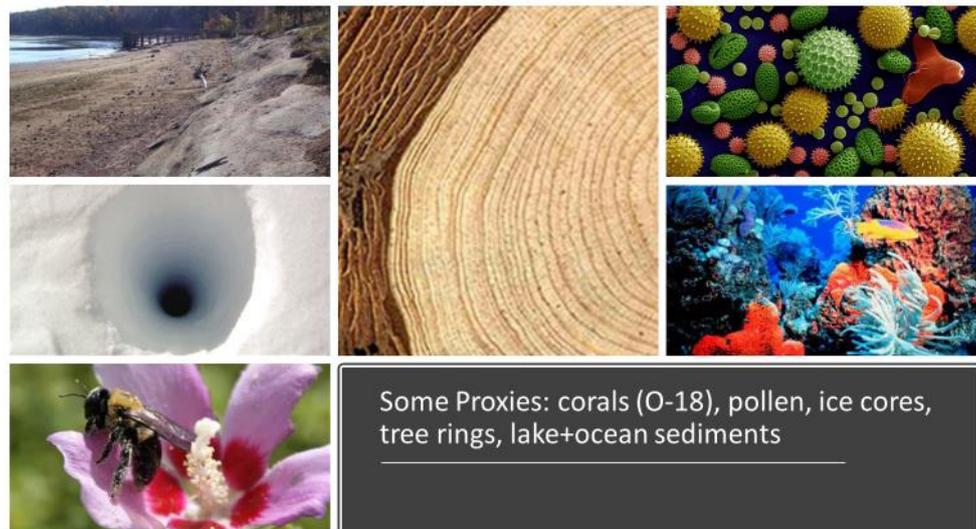


Figure 14. The proxies consist of various natural entities (Liritzis ©).

Paleoclimatologists gather proxy data from natural recorders of climate variability such as tree rings, ice cores, fossil pollen, ocean sediments, corals and historical data (Fig. 14).

By analyzing records taken from these and other (proxy) sources, scientists can extend our understanding of climate far beyond the instrumental record.

Proxy methods are of particular use in the study of the past climate, since direct measurements of temperatures are not available (Fig. 15)

Most proxy records have to be calibrated against independent temperature measurements, or against a directly calibrated proxy, during their period of overlap to estimate the relationship between temperature and the proxy.

The longer history of the proxy is then used to reconstruct temperature from earlier periods (Fig.15).

Proxies are derived from the following environmental data:

- 1) sedimentation rates in lakes (Mintz 2016; Lane et al 2019; Costa et al., 2019)
- 2) quantity variation of pollen use of Pollen as Climate Proxies (Brewel et al., 2007)
- 3) tree rings (Schollaen et al., 2013)
- 4) ice cores<sup>5</sup>
- 5) magnetic susceptibility in lake/marine sediments (Aidona & Liritzis 2012).
- 6) stable isotopes  $^{13}\text{C}/^{12}\text{C}$ ,  $^{18}\text{O}/^{16}\text{O}$  in foraminifera (Mortyn and Martínez-Botí, 2007).
- 7) radioactivity variation per marine/lake sediment depth (Liritzis et al., 1994).

8) The development of Mg/Ca in foraminifera as a proxy for temperature is a perfect example of the development of a new paleoclimate tool (Gray et al., 2019; Bryan and Marchitto, 2008; Henderson, 2002)

9) Historical data Advances in Climate Change Research (Zhang, 2011)

The proxy indices divide in 4 categories. The causes of these are of terrestrial and astronomical origin (Fig.16).

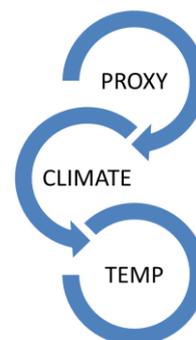


Figure 15. The interlinking of proxies with climate and temperature (Liritzis ©).

These can be summarized as follows:

1. **Glacial:** a) geochemistry of ions and isotopes ( $^{16}\text{O}$  and  $^{18}\text{O}$  and  $^1\text{H}$ ,  $^2\text{H}$ )<sup>6</sup>, b) gas in trapped bubbles, c) trace elements & microparticles, d) physical properties.

2. **Geological:** A) marine: i) biogenic sediments (plankton and benthic fossils), isotopes O, flora, morphological variations, diatoms, ii) inorganic sediments (conti-

<sup>6</sup> The isotopes of particular interest for climate studies are  $^{16}\text{O}$  (with 8 protons and 8 neutrons that makes up 99.76 percent of the oxygen in water) and  $^{18}\text{O}$  (8 protons and 10 neutrons), together with  $^1\text{H}$  (with one proton and no neutrons, which is 99.985 percent of the hydrogen in water) and  $^2\text{H}$  (also known as deuterium (D), which has one proton and one neutron). All of these isotopes are termed 'stable' because they do not undergo radioactive decay.

<sup>5</sup>see:[https://eo.ucar.edu/staff/russell/climate/paleoclimate/ice\\_core\\_proxy\\_records.html](https://eo.ucar.edu/staff/russell/climate/paleoclimate/ice_core_proxy_records.html)

mental (aeolian) dust & fragments of ice, mineralogy of alumina),

B) Terrestrial or mainland: ice deposits, features include glaciers, coastlines.

3. **Biological:** A) rings tree (thickness, density, isotopic composition), b) pollen (type, proportion, absolute concentration), c) microfossils plants, d) corals (geochemistry), e) diatoms, shellfish, lake fossils, f) allocation of modern population (animals and plants).

4. **Historical:** a) texts, b) phenological findings. (See, Haywood et al., 2019)

A historical review of monographs / books the last 30 years include: Waters 1992; Rapp & Hill, 1997; Holliday, 2004; Pollard 1999; Goldberg et al., 2001; Garrison, 2003; French 2003; Wilkinson, 2003; Karakas & Goldberg 2019, while recently geoarchaeology is developed within the cyber-archaeology digital frame (Siart et al, 2018; Levy et al., 2018). In general, the various groups in the World, apply a particular approach in Geoarchaeological research, subject to available instrumentation, research interest and tradition in the institute (Fig.17).

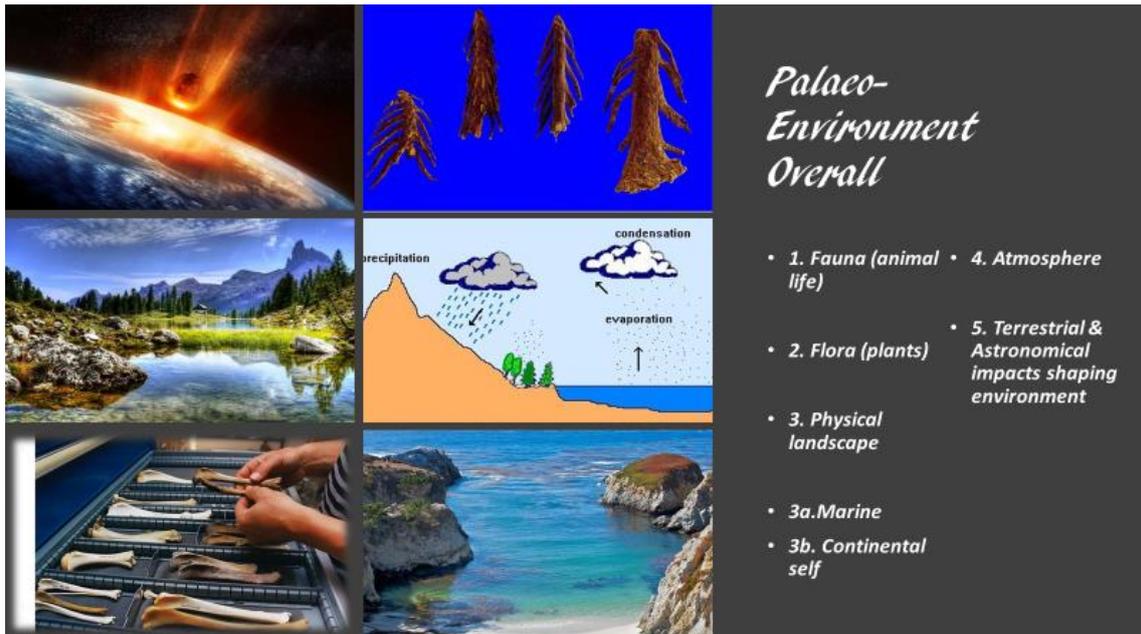


Figure 16. Major factors for the study of the palaeoenvironment (Liritzis ©)

## Approaches in Geoarchaeology

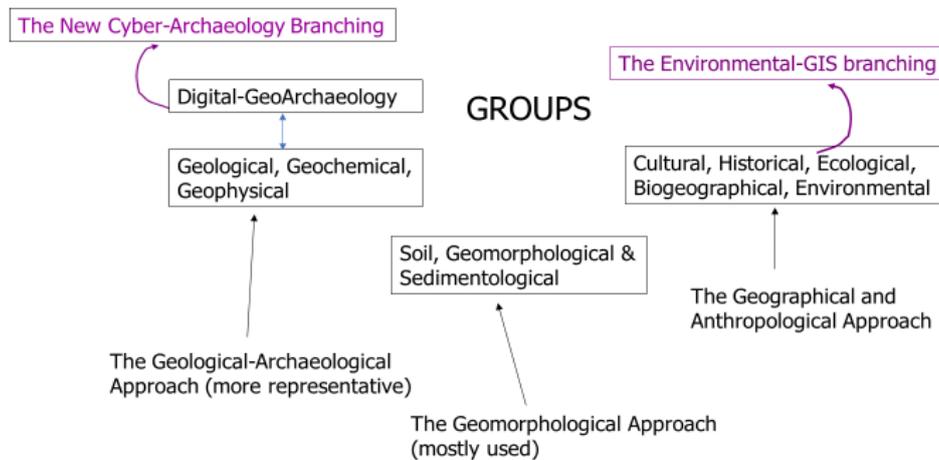


Figure 17. Scientific approaches for geoarchaeological studies (Liritzis © adapted from C.Cordova ppt Oklahoma State Univ).

## 9. CYBER-ARCHAEOLOGY

The term cyber-archaeology introduced into archaeology as a discipline in the late 2000s. Initially was applied to anthropology and communication studies in 1997, where it was used to explain the relationship between computer-mediated communications and online behavior as cultural artifacts (Escobar, et al. 1994). Cyber-archaeology has become by now a branch of archaeological sciences research concerned with the digital simulation of the past. In this context the past is regarded as a generation of the interaction of multiple scenarios and simulations and as the creation of different digital embodiments. The term also recalls the ecological cybernetics approach, based on the informative modeling of organism-environment relationships. The cognitive-interpretive process is accomplished through an interaction feedback loop in a virtual reality environment, following a nonlinear cognitive path (Forte and Danelon, 2019)

The new technologies from virtual archaeology (VA) in cyber-archaeology (CA) and cyber-archaeometry (CAm) are ongoing fast and form an inextricable link to archaeometry. Virtual archaeology is mainly visual, static, with graphics and orientated to photorealism (Reilly, 1990). Recently, new approaches have been added using various interactive practice. The 3D modeling is a very useful practice for the identification, monitoring, conservation, restoration and enhancement of archaeological objects. In this context the 3D computer graphics can support archaeology and heritage policy, offering scholars a "sixth sense" for the understanding of the past, as it allows them almost to live it. CA is the digital management of much partial information in the field. It is not necessarily visual, but dynamic, interactive, complex, autopoietic (self-organized) (Maturana and Varela 1980) and not necessarily oriented to photorealism. The past cannot be re-made, but could be simulated. The CAm is the process of simulation and reconstruction of archaeological finds or cultural materials. The archaeology of the third millennium is capable to process, interpret and transmit much more data and information relative to the last two centuries. CAm is the digital IT process of simulation, restructuring and management of archaeometric processes from the field of natural sciences in relation to material culture, investigated variously (dating, prospection, analysis, technology, provenance, archaeoastronomy, etc.), either as optimum recruited image or as targeted research quest (Liritzis et al 2015; Volonakis, 2019). If this cyber era is seen as a retrospective concept, one has to compare the two approaches in the development of digi-

tal archaeometry from archaeological procedural (processualism) in post-procedural thinking, in order to achieve the analysis of hybrid forms of both approaches, achieved by procedural tools (statistical analysis and quantitative methods in different fields, mathematics, geography, archaeometry, anthropology, archaeology and related disciplines). The virtual computer-aided design (CAD) of all these disciplines is an example of the emergence of Cyber-Archaeometry.

The rapid development of cyber archaeology has led over the past 3 years, to the expansion of informatics tools applied to archaeology and art and to the establishment of new centers (e.g. Center of Interdisciplinary Science for Art, Architecture and Archaeology (CISA3) at UCSD's California Institute of Telecommunication and Information Technology (Calit2), a collaboratory framework has been established facilitating joint research between archaeologists, computer scientists and engineers.

If one sees this cyber era as a retrospective concept, we have to compare the two approaches in the development of digital archaeometry i.e. from archaeological procedure (processualism) to post-procedural thinking. But in order to achieve the analysis of hybrid forms of both these approaches, it requires procedural tools (i.e. statistical analysis and quantitative methods in different fields, mathematics, geography, archaeometry, anthropology, archeology and related disciplines). The virtual CAD of all these disciplines is an example of the emergence of cyber-Archaeometry.

The cyber-archaeology includes: a) the *Virtual Environment* (created on a PC to mimic the real world, b) the *Virtual Reality*, VR, a more specific form of a virtual environment that provides to the user a sense of presence (O'Neil and Perez 2006; Forte, 2000), c) *Massively Multiplayer Online World MMOW* (Virtual Worlds) (a computer-based simulated environment (Bartle, 2003; Aichner and Jacob, 2015) populated by many users who can create a personal avatar, and simultaneously and independently explore the virtual world, participate in its activities and communicate with others facilitated by computers (Bell, 2008), d) *Augmented Reality*, AR (the opposite of the closed world of virtual spaces, a technology that is primarily used in mobile phones and tablets, allows live viewing of a natural environment but whose reality is augmented by viewing information and images of people or places designed through a computer. This is achieved by computer-generated sensory input such as sound, video, graphics or GPS data. Augmentation is conventionally in real-time artificial. (Graham et al., 2012; Rolland et al., 2005), e) *Immer-*

*sive Archaeology* (technology that blurs the line between the physical world and digital or simulated world, thereby creating a sense of immersion (Popper, 2005; Nechvatal, 2005, 2009; Paul, 2008). Here, hardware technologies are developed to stimulate one or more of the five senses to create perceptually real sensations: simulation of senses, vision, auditory, tactile, olfaction, gustation.

Many universities have programs that research and develop immersive technology. Examples are Stanford's Virtual Human Interaction Lab, USC's Computer Graphics and Immersive Technologies Lab, Iowa State Virtual Reality Applications Center, University of Buffalo's VR Lab, and Teesside University's Intelligent Virtual Environments Lab. (Weinberger, 2009; Forte 2010).

f) *Structure from Motion (SfM)* does the first stage of 3D reconstruction. Structure from Motion refers to the method of extracting a 3D structure from many overlapping digital images, and it emerged as a new photogrammetric technique for 3D reconstruction that uses robust computer vision algorithms that automatically detect matching features in images (see an interesting application for the Delphi sanctuary in Liritzis et al., 2016, 2017; Lowe 2004; Ozyesil et al., 2017; Furukawa and Ponce 2007; Hartley and Zisserman, 2003). The more motion and movement around the site, the more complete the 3D model becomes. The collection of matched pixels and their calculated 3D positions become a cloud of millions of 3D points, called a point cloud. The resolution of SfM point clouds is much lower than a LiDAR laser scan, but SfM is much faster, easier to perform, and vastly more accurate than past archaeological methods that relied on surveyors' illustrated plans. The combination of the two technologies allows one to take advantage of both methods strong points, g) *Gamification* (the use-embodiment of various game mechanisms-characteristics, in activities-states not related to game, aiming at the solution of problems via increased users' interactivity and participation (Zichermann and Linder 2013), h) *Serious Games* (Interactive simulations of cultural heritage and museum issues based in game, in which the user participates actively (Anderson et al., 2009; Forte, 2010; Bell, 2008; Liritzis et al., 2015; Maturana and Varela, 1980; Reilly, 1990; O'Neil, and Perez (eds) 2006), i) *Drones and Balloons (UAV)*. Over the past year, increased interest has been developed on the hardware and software required to conduct automated aerial 3D scanning. The Unmanned Aerial Vehicles (UAVs e.g. plane, helicopter and blimp) provide the means to capture images from the air but it is the software used to convert these 2D images into 3D models and merge them with ground scans that makes the technology revolutionary

(Hatzopoulos et al., 2017; Barazzetti et al., 2010; Irshara et al., 2010; Karasik and Smilansky, 2008).

These non-invasive 3D scanning techniques are applied in order to digitally preserve these sites as they are excavated, and to document the inevitable decay of carvings and structures over time, and provide objective datasets for future analysis and visualization. A combination of photogrammetry, SfM, Lidar, UAV, game engine etc, produces significant results for the preservation of cultural heritage, restoration and virtual simulation tasks (Liritzis et al., 2016; Hatzopoulos et al., 2017; <http://ccas.ucsd.edu/>; Vincent et al., 2017).

## 10. CONCLUSION

Archaeometry is a scientifically established international discipline that investigates scientific issues of cultural heritage; it is a multidisciplinary science that develops research and solves archaeological problems. With the help of this interdisciplinary subject new unexplored fields, political, cultural and social landscapes are discovered, and scientific gaps are covered because science, although divided into subgroups, is unified and indivisible.

Archaeometry results consist of data (such as graphs, statistical information, etc.) which simplify and facilitate the possibility of comparing cultural samples and retrieving maximal information from their micro scale, thus conducting safe conclusions, which can be used globally by researchers, scientists and government officials, resulting in the dissemination of information and the globalization of science, the scientific and administrative dialogue, the promotion of administrative functions and the convenience of citizens to fair and proper administrative treatment.

Cultural heritage is without doubt a particularly complex field. The protection and preservation of archaeological sites, ancient monuments, vernacular architecture, industrial installations, cultural and historical landscapes and many other forms of cultural property is and should be the object of many disciplines. The integration of cultural management services with Archaeometry and Information Culture Technologies has already shown the successful impact in many of the processes of documenting and monitoring, interpreting and communicating the data, enhancing many aspects of the research, building capacity and achieving public involvement in the integration of the past into our lives. The management of big data from archaeometrical applications and the cyber-archaeology in the field, the museum, and the office, upraises the interdisciplinary direction to the contemporary new direction of investigation and interpretation of archaeology / anthropology and cultural heritage, the perpetually accredited

scientific holistic approach (PASHA), which has the potential for addressing the new challenges the heritage sector faces and securing its long-term sustainability and preservation, giving a hopeful prosperous future to local, regional and national economic development, from the cultural heritage re-treatment to economic benefit.

At any rate, combined multi-scientific archaeological projects, use “new technologies” (“new” in the sense of updated progressive development in science and technology) and retrieve information in the micro- and macroscopic level. The obtained data of applications to current material culture or in the revealing and documentation of new buried antiquities, with proper interpretation and integration in the wider context of tangible and intangible cultural heritage, are inextricably linked to sustainability.

Archaeometry shows in a scientifically precise, but still palatable and often even amusing way that physical sciences have contributed substantially to the location of buried antiquities, the determination of the age of artifacts, monuments or natural events, the characterization, provenance and authentication of archaeological finds and works of art. Genetic studies - modern and ancient - have become estab-

lished as having the potential to support archaeological investigations with considerable breadth and time-depth. The genes, environment, language and archaeology are individually and together legitimate and pressing subjects of enquiry for the scholar of the past. The improvements in methods, techniques and development of instrumentation aid archaeologists and historians to recognize the potential of science for their purposes, and to gain the knowledge required for the critical evaluation of data provided by scientific methods.

Last, but not least, the virtual archaeology examples over all the World, as a result of advanced technology emerging from computer sciences, stress the naturalistic methodology, challenges and improves digital reconstructions and serious games and at the end offers a holistic approach, taking into account also the value of remote sensing learning outcome and research efficiency. After all, natural and digital methods combined, offer interpretations that is the basis of a synoptic and synthetic philosophy of humans, that merge art, cultural heritage and science, corresponding to classical *techne*, *logos* and *ethos*, for humans.

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