Archaeomineralogy of prehistoric artifacts and gemstones

/ Salvador Domínguez-Bella

Dpto. de Ciencias de la Tierra. Universidad de Cádiz. Campus Río San Pedro. 11510 Puerto Real (Cádiz), Spain

Abstract

The study of the compositional nature, geological and geographical origin of the tools and jewellery used by man in prehistoric times has been since the nineteenth century the scientific goal of some researchers in the fields of mineralogy and petrology. This interest in heritage research studies is experiencing a significant growth in recent decades, with a great development of interdisciplinary collaborations, both from the field of archeology as for the conservation, restoration and management of artistic and cultural heritage. The application of physico-chemical techniques, common in studies of mineralogy, petrology and analysis of materials to the resolution of the fascinating questions posed from the archaeometry and the fact that we dispose of a growing number of analytical techniques with higher experimental performance make that this line of research has grown in the interest of researchers. Here several studies worldwide about some of the most widely used mineral substances throughout history and in different geographical areas and the tools and prestige objects manufacture, used by human societies are summarized. Finally, we present several examples of archaeometric studies carried out on minerals and fossil resins, used during the Prehistory of the Iberian Peninsula, western France and North Africa in the elaboration of tools, jewellery and objects of prestige.

Resumen

El estudio de la naturaleza composicional y del origen geológico y geográfico de las herramientas y joyas usadas por el hombre desde la Prehistoria, ha constituido desde antiguo, el objetivo científico de algunos investigadores de las áreas de mineralogía y petrología. Este interés por los estudios patrimoniales está experimentado un gran crecimiento en las últimas décadas, con un mayor desarrollo de las colaboraciones interdisciplinares, tanto desde el campo de la arqueología como de la conservación, restauración y gestión del patrimonio artístico y cultural. La aplicación de técnicas físico químicas, habituales en los estudios de mineralogía, petrología y análisis físico-químico de materiales, a la resolución de las apasionantes incógnitas planteadas desde la arqueometría y el hecho de disponer de cada vez mayor número de técnicas analíticas y con mayores prestaciones experimentales, hacen que esta línea de trabajo haya crecido en el interés de los investigadores. Se resumen varios de los estudios a nivel mundial sobre algunas de las sustancias minerales más utilizadas a lo largo de la historia y en diferentes áreas geográficas, en la elaboración de herramientas y objetos de prestigio, usados por las sociedades humanas. Finalmente se muestran varios ejemplos de estudios arqueométricos realizados sobre sustancias minerales y resinas fósiles, utilizadas durante la Prehistoria de la península Ibérica, el oeste de Francia y el Norte de África, en la elaboración de herramientas, joyas y objetos de prestigio.

Key-words: *mineralogy, archaeometry, cultural heritage, Iberian Peninsula, raw materials, prehistoric jewels, conservation.*

1. Introduction

The archaeomineralogy is itself a mineralogical sub-discipline with a history of not very long tradition, at least in the Iberian Peninsula. This specialization of mineralogy has been developed in parallel to archaeometry studies applied to materials in archaeological and artistic heritage. The first reference to this term appears in *Mitchell, 1985* and contrary to the disciplines of Geoarchaeology, has not yet had recognition as such, although in recent years there has been a large increase in the interest of mineralogists in the study of archaeological materials *(Turbanti Memmi et al. 2011)*.

A current definition of archaeomineralogy appears in *Rapp (2003 and 2009)*, as "the study of minerals and rocks used by ancient societies across space and time, as tools, ornaments, building materials and raw materials for metals, ceramics and other processed products".

In recent years archaeomineralogy studies have been increasing. This may be due to several factors:

- Increase in interdisciplinary studies in archeology, with the participation of many specialists from different scientific disciplines *(Price & Burton 2011)*, including among them the mineralogy and petrology.
- Higher number of analytical techniques available for studies of mineralogical and geochemical characterization of the samples for study and greater analytical precision and capacity of them.
- Increased interest from the field of archaeology and restoration of historic and artistic heritage in the potential of these techniques on obtaining more and better archaeological and historical information.

2. Application of mineralogical studies in different archaeological problematic

Mineralogical disciplines provide valuable information to design solutions to many of the problems, from an archaeological point of view, for the study of historical and artistic heritage and the restoration of works of art and monuments.

However there are problems inherent in developing partnerships, like this:

- From the mineralogy and geology in general, it is possible to have a perception of materials, types and natural processes that originated them within a broader regional geological context, so that the background in geology and mineralogy should be considered a fundamental and necessary part, both for research archaeometric to those relating to heritage conservation. So we need a good understanding of what have been the physical-chemical systems and processes involved in the genesis of each mineral or rock.
- Besides it is particularly important that there is reciprocity in the transmission of data among mineralogists and scientists in general, working on heritage and humanistic counterpart specialists, this is archaeologists, museum and collections curators and cultural heritage managers *(Artioli & Angelini 2011)*. This interdisciplinary collaboration, consistent and objective, will provide a complete picture of the issues to be addressed in each case and will avoid producing an "innocent archaeometry" *(Ramos et al. 1998)* in which the analytical data are presented only as annexes to the archaeological work and very often with no connection between the conclusions of both.

Recent contributions as book edition of Rap (2009) on archaeomineralogy or monographic works as the one published in the European Society of Mineralogy *(Turbanti Memmi et al. 2011)* show the growing interest in this line. Many minerals and rocks as prehistoric artifacts used as gems in antiquity have been studied archaeometrically in recent decades.

The variety of compositions, colors and geological origins that these present is very significant and there are many archaeological problems that are related to these lithologies and raised by archaeological research, in some cases for a long time *(Damour 1864)*. This is equally applicable to other minerals used as pigments since prehistory to the present *(Domínguez-Bella 2010a; Domingo et al. 2012)*.

Very different mineralogical techniques have been implemented for its resolution in the last century, due to the evolution of technological and analytical capacities for its identification and characterization, in constant evolution, especially in recent decades *(Artioli 2010)*.

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3. Analytical techniques at the archaeometry of prehistoric artifacts and jewellery

Different work strategies can define, such as combined techniques (ie, the analysis techniques applied to the same object at different times) and / or simultaneous (ie, several analytical techniques are applied to the same object simultaneously), since in many cases, a single technique is not sufficient by itself to resolve complex problems.

The pre-treatment of the samples and the amount of the same required for analysis varies depending on the analytical technique to be employed as well as its nature. In general for non-invasive techniques, samples can be analyzed without any preparation *(Fig. 1 & 2)*, while in other more aggressive as the polarized light microscopy the required amount of sample is generally greater. In most cases, samples can be prepared quite easily, as occurs in many geological laboratories available in research centers or companies.

Today a great effort is being focused in the development and optimization of portable

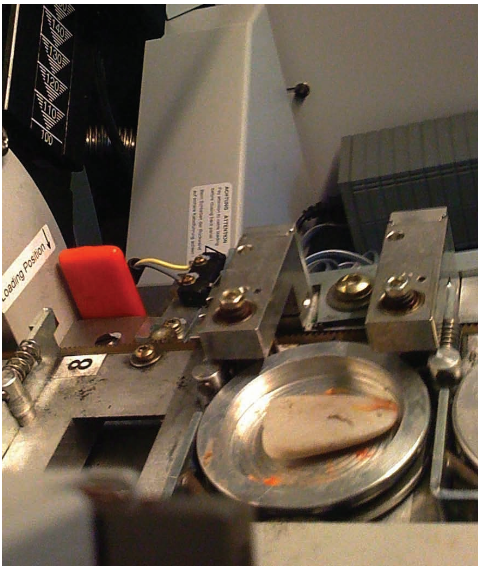

Fig. 1. Direct XRD analysis of archaeological objects. Sillimanite axe from a Neolithic site of Cadiz province, SW Spain. SCCYT, Cádiz University

Fig. 2. Sample-holder for direct and non-destructive WDXRF analysis of a sillimanite adze from the Neolithic of Brittany (France). SCCYT, Cádiz University

instruments in order to perform in-situ analysis, in museums, institutions and the field, including XRF spectrometers, optical and Raman sensors, X-ray diffractometers and other instruments. Scientific and protection of cultural world heritage institutions have been working hard in the last years to have laboratories capable to make fast and cheap analysis. Despite this, it is especially important to perform a study and previous programming of the technique or techniques that are most suitable for the study of a particular type of material and to have a knowledge of the protocols for measurement, the instrumental configuration, procedures calibration, and ultimately, optimization and suitability of each technique or set of techniques with respect to the nature of the material to investigate.

We currently have a wide range of analytical techniques with more or less invasive in relation to the amount of sample affected, although many of them, traditionally destructive sample preparation, can be applied on a non-destructive and analytical results quite reliable. This would be the case, for example, the X-ray diffraction (XRD) and X-Ray Fluorescence diffusion wave (WDXRF), applied to archaeological materials of small size and with a flat surface *(Figs. 1 & 2)*.

Among the usual techniques in archaeometry we can mention: Optical microscopy (MSC), Polarized light microscopy (PLM), X-Ray Diffraction (XRD) (powder or direct method), X-Ray Fluorescence diffusion wave (WDXRF), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy with Energy Dispersive X-ray Analyzer (SEM-EDX), Raman microscopy (RM), Optical Emission Spectroscopy (OES), Induced Coupled Plasma Mass Spectrometry with laser Ablation (ICP-MS-LA), Inductively-coupled Plasma Optical Emission Spectroscopy (Laser Ablation) (ICP-OES-LA). Also other techniques reported as: Laser microprobe mass analyzer or Laser induced mass analyzer (LAMMA / LIMA), Atomic absorption spectrometry (AAS), Flame atomic emission spectroscopy (FAES), Atomic-emission spectroscopy (AES), Instrumental neutron activation analysis (INAA), Electron diffraction (ED), ion probe (secondary ion mass spectrometry) (SIMS), radiometry, emission spectroscopy, spectrophotometry, etc.

Many of these or other qualitative, semiquantitative or quantitative techniques, classified as nondestructive analysis techniques are: Optical microscopy with visible light (OM), Cathodoluminescence microscopy (CL), Ultraviolet microscopy (UV), Infrared microscopy (IR), IR absorption spectrometry (Fourier transform IR microspectrometry) (FTIR/FTIRM), Laser Raman microprobe (or Laser Raman spectroscopy) (LRM/LRS), Portable X-ray Fluorescence (PRXF), micro X-Ray Diffraction (µXRD) Electron microprobe (EPMA), Transmission electron microscopy (TEM), Synchrotron X-ray fluorescence (SXRF), X-ray absorption fine structure (XAFS), Prompt Gamma Activation Analyses (PGAA), Elastic recoil detection analysis ERDA, Extended X-ray absorption fine structure (EXAFS), Nuclear magnetic resonance/Proton magnetic resonance (NMR/PMR), etc.

An important role is played by isotopic (Carbon-Oxygen-Sulfur) determination techniques, especially in provenance of, for example, metamorphic rocks as marbles or in minerals as cinnabar used as pigment *(Minami et al. 2005).*

The experimental parameters and the features and availability of each of these techniques are very different, and its use alone or in combination, depending on the type of substance and the specimen to analyze.

4. Prehistoric Artifacts

Many examples of minerals and rocks used since the beginnings of prehistory for the elaboration of different kinds of artifacts and tools can be mentioned.

We emphasize the use of silicate minerals and rocks, and metamorphic rocks in relation to mineralogical and petrological studies of minerals and rocks used since the Palaeolithic *(Domínguez-Bella et al. 2010)*, such as quartzite, volcanic and plutonic rocks and sedimentary rocks as silicified sandstones, the group of flint and radiolarite *(Navazo et al. 2008)*, as well siliceous minerals as microcrystalline quartz varieties (agate, carnelian, onyx, etc.), single crystals of rock crystal *(Domínguez-Bella and Morata 1995)*, volcanic glass as obsidian *(Tykot 2002)* or organic substances such as fossil resins *(Beck et al. 1964 & 1965),* etc.

There is a extensive bibliography on the characterization and identification of source areas for these materials based on the use of many analytical techniques, including some classical techniques predominant in the geological sciences as OM, XRD, XRF, and more specific as RM, FTIR, ICP-MS-LA, PIXE, PIGE, INAA, µXRD, PXRF, CL, Carbon-Oxygen-Sulfur-Strontium isotopes, etc. These specific techniques vary depending on the substance under consideration and the technological development of new analytical techniques in recent decades *(Price & Burton 2011)*.

Through these studies, we have nowadays great information on mobility of raw materials in prehistory, such as obsidian in the central basin and the western Mediterranean *(Tykot 2002)*, in Central Europe *(Rosania et al. 2008)* or in Mesoamerica and South America *(Jiménez-Reyes et al. 2001; Rivero-Torres et al. 2008; Tenorio et al. 1997; Seelenfreund et al. 2005)*; of siliceous materials as flint and radiolarite in Europe by using more or less sophisticated techniques as AAS, XRD, ICP-MS and ICP-AES *(Navazo et al. 2008), or* with simple microfacies studies with MO *(Della Cassa 2005)*.

The bulk of the analytical work on polished rocks, has taken place on the most common lithologies in the Neolithic and Chalcolithic, which have a regional or continental distribution from production centers, such as the axes of prestige elaborated in jadeite and HP green rocks of alpine origin *(Petrequin et al. 2012; Cassen et al. 2012; D'Amico et a.l 2003)*, or the dolerites of Plusulien, France *(Le Roux 1999)*, the amphibolites in the West of Iberian Peninsula *(Lillios 1997),* hornfels in NE Spain *(Risch & Martinez 2008; Clop 2004)*, sillimanites-fibrolites of the Iberian Peninsula and France *(Goer de Herve et al. 2002; Domínguez-Bella et al. 2004; Aguayo de Hoyos et al. 2006; Pailler 2009)* or flint from Casa Montero, Madrid *(Bustillo et al. 2009)* or Benzú, Ceuta *(Ramos et al. 2008).*

Rocks formed under conditions of high pressure (HP), deserve a special attention such as green rocks as jadeite, eclogite, etc., which have been used in different periods and geographical areas since prehistoric times *(Ruvalcaba et al. 2008; Cassen et al. 2012)*, often polished and with a high value as prestige goods.

Mineralogical, petrological and geochemical study techniques have been used for the classification and determination of their source areas *(D'Amico et al. 2003; Sheridan et al. 2010).*

4.1. Archaeomineralogy of prehistoric artifacts, examples in the Iberian Peninsula, West of France and North Africa

From the end of the XIXth century to the beginning of XX, geologists worked with the prehistorians in the examination of archaeological materials that composed these lithic elements to attempt the characterization of the constituent rocks and their geological and geographical origin. The works of Quiroga (1885) and of San Miguel de la Cámara (1918) were the pioneer studies in mineralogy and petrography of archaeological objects in Spain.

But these petrographical studies were not continued for many reasons since this information could not be correlated with that one given by the geological outcrops, mainly because the basic geological works had not been done, which would have given an adjudication of the analyzed rock type with a definite geological outcrop. At that time, works of geological cartography were beginning to start, with the creation of the Commission of the Geological Map of Spain. On the other hand, in those days, a great number of prehistorians still considered more important the object itself than its archaeological significance.

To these obstacles we also have to add the fact that the analysis methods were expensive and destructive. It is not till the 80 and 90 decades of the XXth century that more or less systematic petrographic studies will return, in this type of materials in Spain. *(Domínguez-Bella y Morata 1995; Domínguez-Bella et al. 2004).*

In recent years archaeomineralogical studies have been ongoing on archaeological materials abiotic prehistory, especially the so-called stone industry, these materials can separate two groups, industry and polishes carved.

The study of the lithic industry has a strong mineralogical and petrographic and geochemical component, so that participation of these disciplines is very important to determine the mineral nature of the object and their petrographic, paleontological and geochemical features. These factors are of great interest both from the determination of the source area of these raw-materials and the features and physical properties of the rock.

The determination of the source areas of mineral raw materials is one of the main issues of concern archaeomineralogical studies, these studies allow to obtain great archaeological information, both on the strategies for obtaining of lithic resources, by prehistoric societies, their exploitation techniques if they exist (underground mining for example) *(Camprubí et al. 2003; Bustillo et al. 2009)*, mobility of these groups in the territory, the use of the lithic material and determination of transport over short, medium or long distances, in case there are organized networks for such distribution, as occurs with some precious or exotic materials, which can travel long distances (up to thousands of kilometers), from its geological source areas to where they are deposited *(Domínguez-Bella et al. 2002; Cassen et al. 2011; 2012; Querré et* *al. 2012; Rubalcaba et al. 2008)*.

The study and determination of mineralogical and textural properties of many rocks and minerals is also of archaeometric interest, as these properties can be direct determining factors for a particular use by prehistoric societies of the material. These first steps in the "materials science" are interesting examples in many of the studies currently being developed, such as lithic assemblages in the Palaeolithic and Neolithic, indicating high levels of technical knowledge, obtained certainly through the experimentation. By means statistical analysis applied to mineralogical-petrological classifications of tools and their relationship with their technological type, is verified in many cases a deliberate selection of certain raw materials for a particular use, such as flint and radiolarite in the Palaeolithic environment of the Strait of Gibraltar as Embarcadero of Palmones river, Algeciras or Benzú rock-shelter, Ceuta *(Domínguez-Bella et al. 2004)*.

In the studies that we are doing since 1994 on these materials, in southern Spain and northern Africa it has been shown or inferred allochthonous or autochthonous provenance *(Domínguez-Bella and Morata 1995; Domínguez-Bella, Perez and Morata 2000; Ramos and Giles 1996; Domínguez-Bella et al. 2000, 2004 and 2006)*.

Within the group of analyzed knapped stone materials, many minerals and rocks, especially siliceous, constitute in percentage the main group in prehistoric lithic industry. These include flint, radiolarites and jasper; we can join with other siliceous sedimentary and metamorphic rocks as silicified sandstones and quartzites *(Hernández et al. 2012)*.

4.2. Metamorphic rocks in the Atlantic Band of Cádiz, SW Spain

4.2.1. Sillimanite/fibrolite, amphibolites, marbles

The sillimanite-fibrolite (Al_2SiO_5) is a metamorphic mineral of high temperature and variable pressure, which appears in high-grade metamorphic rocks. This is not an exceedingly rare mineral in metamorphic environments, although it is more limited when it comes to centimeter or decimeter-sized nodules. This relatively wide distribution in metamorphic areas makes it difficult to define the source area of the samples, usually very homogeneous in macroscopic appearance.

The size of the nodules of sillimanite seems to be a determining factor when a geological outcrop is susceptible of constitute a source area for the manufacture of polished stone tools of sufficient size.

Polished tools of these lithologies, are widely represented in the recent prehistory of the Atlantic Band of Cádiz and other peninsular areas and of the Northwest of France, the problem of their possible origin is still open to new theories and analysis, even in progress. In the case of SW Spain and Portugal, we think that sillimanites can be completely allochthonous materials to this region, given the scarcity and small size of sillimanite nodules that appear in the Betic Cordilleras. However some small outcrops have appeared with this mineral in the province of Malaga (*Aguayo de Hoyos et al. 2006)* that according to these authors could be the source area for the productions in the area.

We are currently working on the possible determination of the source areas of this mineral, relating the geological material analysis of the most important sites in the Iberian Peninsula, North Africa and France, as are those of the province of Segovia, Madrid and Avila (Sierra de Guadarrama), points of the Serranía de Ronda, Sierra Morena, west peninsular zone (Zamora, Salamanca), Brittany and Massif Central (France), Tetouan area (Morocco), among others.

Within these lithologies, generally scarce in the southern Iberian geological environments, the most important in terms of their proportion in the archaeological record in the Atlantic region of Cadiz are amphibolites, some metamorphic rocks such as marbles and quartzites and a few volcanic as tuffs *(Domínguez-Bella et al. 2004)*. Some of these exotic lithologies would be possible source area, outside the scope of the Betic Cordilleras. In the Iberian Peninsula there are several places you could find amphibolites similar to those described in the archaeological record of the Atlantic Band of Cádiz. The possible source areas closer to the area would be in volcano-sedimentary sequences, the southwest sector of the peninsula, particularly in the Ossa-Morena zone (provinces of Huelva, Seville, Badajoz and Alentejo area, south Portugal) and the amphibolites, the source could also be in the zone north of Huelva, Seville and south of the province of Badajoz *(Domínguez-Bella & Morata 1995; Domínguez-Bella et al. 2004)* and various points in Portugal *(Lillios 1997)*. A future line of work should address the petrological, geochemical and mineralogical different outcrops of these rocks in the western peninsula and the Betic Cordillera in order to be able to establish a database that allows discrimination of different exposures and allow the determination of -source areas for these archaeological materials widely distributed in recent prehistory, of which there is already evidence that were exploited by means open pit quarry, somewhere in the western peninsula.

Regarding the articles made of marble, they highlight the bracelets in one piece, made by turning from fragments of this rock type and relatively common in the Late Prehistory of the area. We have analyzed some significant records in areas bordering the Atlantic Band of Cádiz, including: El Jadramil (Arcos de la Frontera) (*Domínguez-Bella 2003)*, Ardales (Málaga) (*Domínguez-Bella et al. 2001 and 2004)* and Villamartin, Cadiz. Its origin has not yet been determined analytically.

4.2.2. Serpentinites and peridotites

In this type of ultrabasic rocks we have studied some objects recovered in the archaeological record of the area around the Strait of Gibraltar. They are usually colored beads made in dark green serpentine, as in the site of Cantarranas-La Viña *(Domínguez-Bella, Perez & Morata 2000)* with an allochthonous origin, due to the absence of these rocks in the region. Other site is the cave of Benzú (Ceuta), placed in north Africa *(Chamorro et al. 2003 & Domínguez-Bella et al. 2006)*, where the origin of these materials is local, since there is an outcrop of ultrabasic rocks in the city of Ceuta, close to the cave, which may be the source area of these materials currently under study geochemical and mineralogical XRD-XRF and MO.

5. Prehistoric Jewellery

The wide variety of minerals and organic materials used as luxury or prestige jewellery *(Bard 1999)*, their geological origin or source areas, exploitation and trade routes of transportation and exchange, make these studies from the analytic sciences of great interest for archaeologists and museum and collections curators. It is one of the most exciting in the archaeomineralogy *(Guillong & Günther 2001; Kosmowska-Ceranowicz 1990 & 2003; Domínguez-Bella 2004; Ruvalcaba et al. 2008; Querré et al. 2012; Calligaro et al. 1998 & 1999)* and one of the archaeological materials that has attracted more interest from the archaeometric point of view, the gemmological minerals and substances.

There are interesting examples in the human historical record since the Palaeolithic, but a special abundance of these objects appear in the recent prehistory of Europe and Africa (Neolithic-Chalcolithic) or pre-Columbian times in America. Gemmological materials have been widely distributed over large commercial networks throughout history, as in the case of rubies, sapphires and emeralds, etc., highly appreciated in Roman times and later *(Calligaro et al. 1998; 1999; Aurisicchio et al. 2005; Giuliani et al. 2000)*.

Notable examples in the minerals used in jewellery may also be some green minerals from prehistory which have been a constant across cultures and geographies.

They highlight examples such as jade in Mesoamerica or Asia *(Casadio et al. 2007)*, turquoise in Mesoamerica and South America *(Domínguez-Bella & Sampietro 2005; Hull et al. 2008), in Asia in the west Europe (Vázquez Varela 1983; Domínguez-Bella 2004; Querré et al. 2012*).

From among the green mineral, as well as other colors, deserves special attention jade, a material highly valued since prehistoric times in different cultures, chronologies and geographical areas. During the last decade

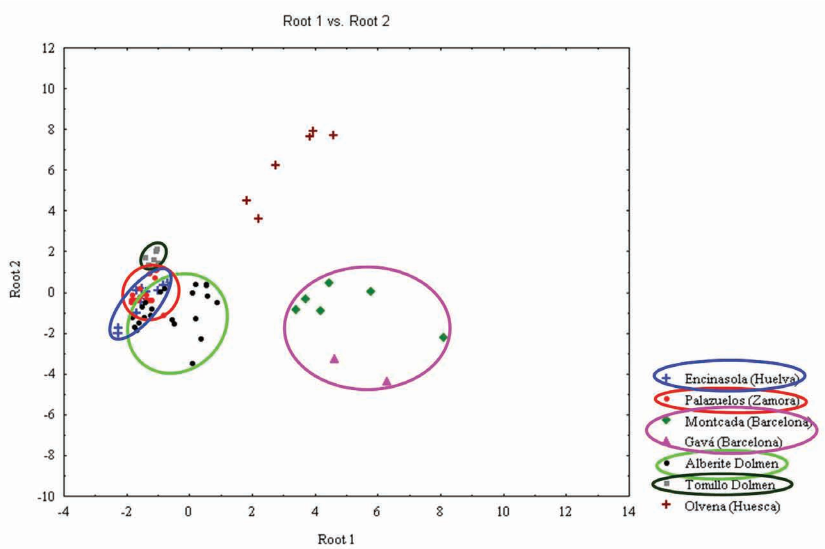

Fig. 3. Multifactorial analysis distribution in geological and archaeological Neolithic variscites from ICP-MS-LA geochemical data (Domínguez-Bella et al., 2002).

different methods of analysis of this mineral substance using a wide range of analytical methods have developed to determine their geological sources, early jade working methods, the detection of heating processes in jade, burial and surface alterations.

Of particular interest is the use of non-invasive Raman microscopy (RM) in the study of Mesoamerican jadeite pebbles from Guatemala *(Gendron et al. 2002),* This application of RM to jadeite could become a routine approach in archaeometry for identification and provenance studies, especially as inexpensive portable Raman microprobes are developed with improved spectral resolution (up to 8 cm^{-1}).

Other techniques as X-ray fluorescence spectroscopy (XRF) and external beam particle-induced X-ray emission (PIXE) have been applied to the study of jades. Chinese jades dating from the Neolithic period (5000 to 1700 BCE) to the Han dynasty (206 BCE to 220 CE), composed of nephrite, has been analyzed in order to determine the minor elemental compositions of these objects, and their geologic source in China *(Casadio et al. 2007).*

Many analytical techniques already cited are being used in the study of prehistoric jewellery, especially those of greater use in the field of mineralogy as OM, XRD, WDFRX, PXRF, RM, CL, PIXE, PIGE, etc. Also of interest are some analytical techniques for the study of fluid inclusions, especially important in some of the gems, in relation to their genetic and therefore its source and origin area. A synthesis of specific analytical techniques for these studies has been described by *Anderson & Mayanovic (2003).* The same occurs with some applications of isotope studies in determining the source areas in gems *(Giuliani et al. 2000).*

5.1. Archaeomineralogy of gemstones in the Prehistory of Iberian Peninsula, France and North of Africa

While the Iberian Peninsula and in general, south-western Europe, are not rich in gemmological materials, there are some exceptions that have been of great importance in the exploitation, processing, transportation and distribution of minerals and rocks used in the production of precious or prestige objects, not always gems, along prehistory in this geographical area.

Minerals of the silicate group are also common in prehistoric jewellery, so we can find examples similar to the Near East steatite *(Allen et al. 1975),* such as beads of talc, clinochlore and micas in many different prehistoric sites in the Peninsula, where they appear as pendants or beads necklace. There are many examples in the Iberian Peninsula in recent prehistory sites as Katillotxu dolmen in Biscay *(Quintana 2009)* or the site of Leceia, Portugal *(Cardoso 2002)*. Other silicate minerals such as clinochlore have been analyzed in recent prehistoric sites, as in the tumulus of the Higueras Valley, Toledo *(Domínguez-Bella 2010)*.

Depending on the type of material, its composition and origin have many different analytical techniques employed in the work of archaeometric characterization carried out in recent 50 years. Thus, for the majority of silicate compounds, oxides, sulfides, phosphates, carbonates, etc., has been used traditional techniques such as XRD, XRF, OM, FTIR and in recent years, especially in samples belonging to the funds of museums and collections, new non-destructive techniques such as PIXE, PIGE, XR microdiffraction, RM, EDX, ESEM, LIBS, PXRF, etc., are being used.

Techniques such as mass spectroscopy, inductively coupled plasma, laser ablation (LA-ICP-MS) *(Domínguez-Bella et al. 2003)* that can provide a detailed geochemistry of the samples and that together with statistical studies of factorial analysis of data have allowed us to identify possible source areas of origin of products such as archaeological variscites *(Domínguez-Bella et al. 2002) (Fig. 3)*. The same occurs with techniques such as PIXE, PIGE that we are applying to these phosphate minerals within the project CALLAIS, CHARISMA program, in development since 2010 and using the facilities of AGLAE in the Louvre, Paris *(Fig. 4)*. We are currently working on multivariate statistical treatment of data obtained over a wide sampling of variscitas and turquoise of the Iberian Peninsula and France, as well as archaeological samples from all known geological sites in Spain, Portugal and France, with the collaboration of Museums as Huesca, Braga, Bilbao, British Museum, etc. Some of the results of these and previous analyses have been published *(Querré et al. 2012)*.

The X-ray fluorescence (WDXRF) and portable (PXRF) also allows a quite precise analytical, especially for the major elements in the sample and with a non-destructive character *(Domínguez-Bella & Bóveda 2011)*.

Fig. 4. Variscite necklace beads from the Neolithic site of Saint Michael, Carnac, France. Non-destructive analysis by PIXE-PIGE. AGLAE, Louvre Museum, Paris. CALLAIS project.

5.1.1. Variscite and turquoise

Another green mineral of interest is the variscite ($Al_2O_3PO_4 \bullet nH_2O$), which geological rarity and interest by the Neolithic man is a clear example of precious material in prehistory. In Europe there are not many geological areas containing this phosphate, usually associated with its isomorphic variety, the strengite ($FePO_4 \bullet 2H_2O$) and sometimes other phosphates such as turquoise ($CuAl_6(PO_4)_4(OH)_8 \bullet 4H_2O$) *(Moro et al 1992a, b, 1995b)*. Underground mining techniques for selective procurement of this mineral are well known in Gavá, Barcelona *(Fernández-Turiel et al 1990; Camprubí et al 2003)* and distribution of this material in the N and NE of the Iberian Peninsula (*Munoz-Amilibia 1971; Guerra et al. 1995; Fernández Vega & Pérez Cañamares 1988; Edo et al. 1998)* and the SE of France *(Villalba et al. 1998)*.

Mineralogical and geochemical analysis by ICP-MS-LA, XRF, etc., of these precious objects in phosphate minerals were used to determine their source and distribution areas

Fig. 5. Selection of objects made from jade, fibrolite and variscite from the cist at Saint-Michel (photos: S. Cassen and C. Le Pennec, collection of the Soc. Polymatique du Morbihan, musée de Vannes).(in: Cassen et al., 2011) and in: www.jungsteinSITE.de

(Guerra et al. 1995; Domínguez-Bella et al 2002 & 2003).

The variscite appears among others in necklace beads recovered from the burials of the dolmens of Alberite I (V and IV millenniums BC) *(Domínguez-Bella & Morata 1995)* and Tomillo *(Domínguez-Bella et al. 2002)*, (IV-III millennia BC) in the province of Cadiz. It is a green mineral which has had a great importance in Neolithic-Chalcolithic societies in southwest Europe.

In this area and many others of the peninsula and in some places of France, it is relatively frequent the appearance of objects elaborated in this mineral, usually necklace beads, which in the dolmen of Alberite represented 7% of over recovered 1000 necklace beads.

The XRD study of some of these beads revealed a monomineral nature, corresponding with the type variscite Palazuelos. IR spectra of these samples showed absorption bands characteristic of the molecular groups $(OH)^-$ and $(PO_4)^{3-}$, coinciding also with the variscite.

These beads have a similar mineralogy, and microscopic examination shows that it is generally monomineral samples, massive, fine-grained, pale green, colorless, non pleochroic and low relief, interference colors of the second order. The qualitative chemical analysis performed by means EDX as expected, showed the presence of P and Al.

The green beads of these chronologies present different mineral compositions (variscite, turquoise, talc, muscovite), with different geological origins and many times geographical *(Damour 1864; Cardoso 2000; Fernandez Vega & Cañamares Perez 1988; Huet B. Gonçalves 1980 & 1982; Muñoz-Amilibia 1971; Rojo et al. 1995; Querré et al. 2012).*

It seems that the green color of these minerals was the main feature wanted by these communities for the elaboration of these objects, whatever their composition, hardness, etc. The green color should be a sign of prestige or some ritual significance, highly desired by the dominant members in the communities. This idea is shown by the fact that it is only in burials of the dominant groups in which the large necklace beads and pendants of these minerals appear, as in the megaliths of French Brittany *(Cassen et al. 2011 & 2012)*, Galicia *(Dominguez-Bella & Bóveda 2011)*, Portugal, Extremadura and Andalusia, or the pit burials Culture in Catalonia *(Domínguez-Bella 2004)*.

The variscite and turquoise beads are associated in these funerary environments *(Fig. 5)* with other minerals that also had to have a significance of prestige or ritual, such as cinnabar, amber, rock crystals and large flint blades and axes, chisels and adzes polished rocks, idols and palettes for pigments *(Ramos and Giles 1996; Cassen et al. 2011)*.

The emergence and expansion of variscite necklaces become important in the Neolithic south-western Europe from the sixth millennium BC and their use lasts until the Roman Empire, where it is mined in western Spain, replacing and / or imitating emeralds.

The variscite outcrops in south-western Europe with special geological features are found almost exclusively in the Iberian Peninsula. Vein deposits of this mineral, associated with Silurian slates with black siliceous levels and quartzites, appear in the Palaeozoic of North Portugal (Ervedosa), the provinces of Zamora (Palazuelo de las Cuevas, Bercianos, El Bostal, etc.) and Huelva (Encinasola), Galicia (Punta Montalvo)

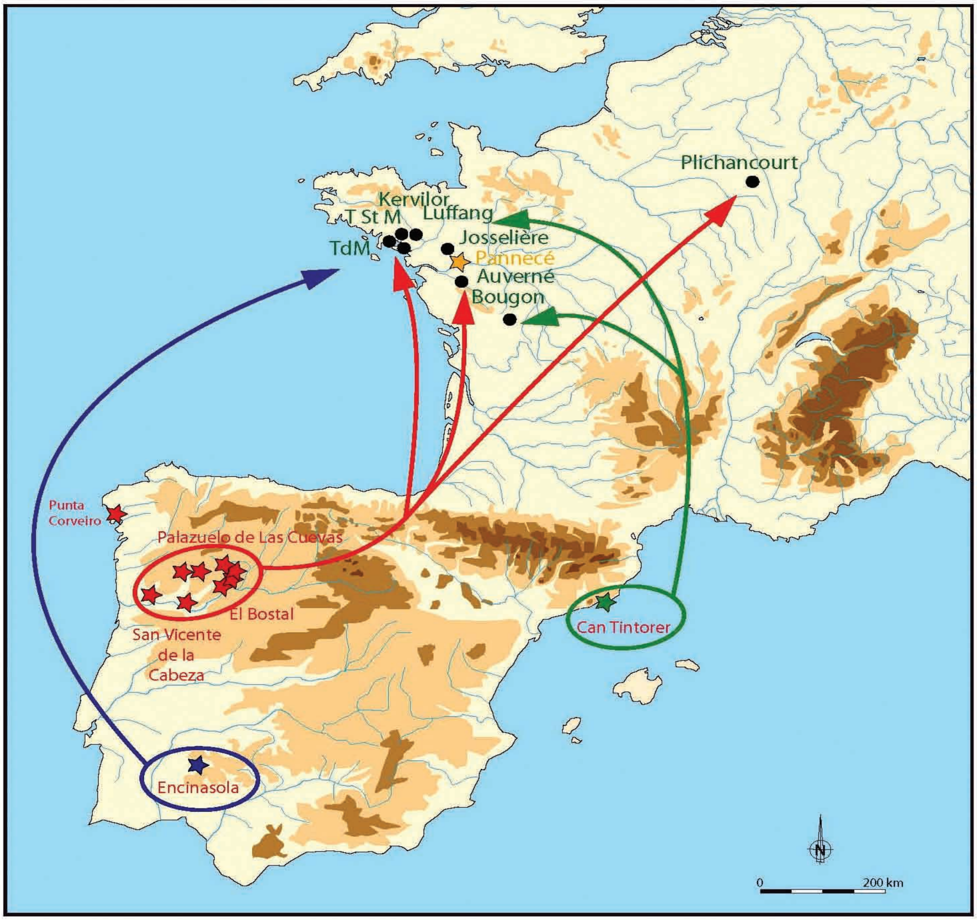

Fig. 6 Large distribution networks over long distances for Iberian variscites have been identified from these sources in the French Brittany (Querré et al., 2012)

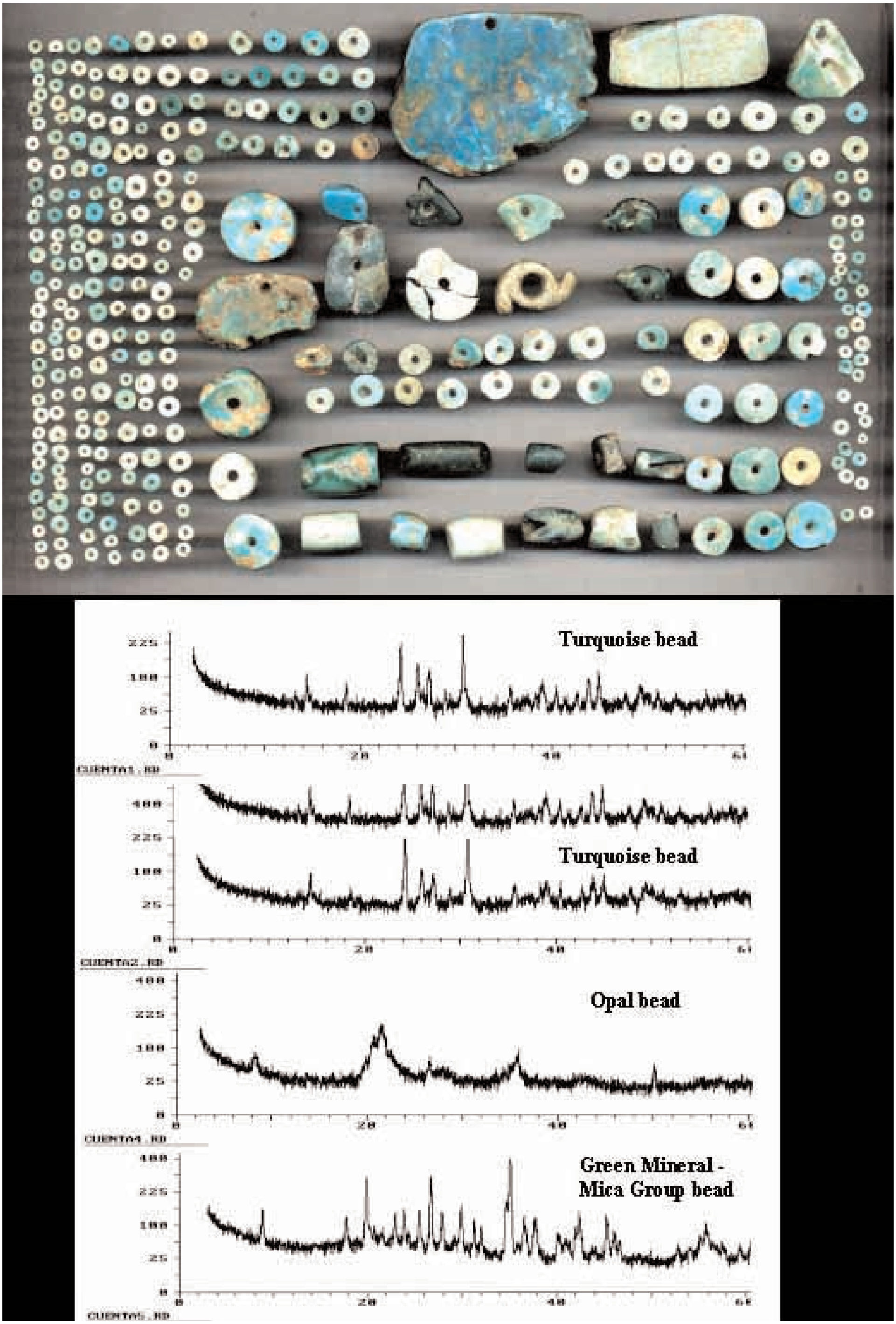

Fig. 7. Selection of turquoise beads from the Tafí Culture, Tucumán province, Argentina and direct XRD diagrams of the representative lithologies of the beads (turquoise, green mica, opal). (Domínguez-Bella & Sampietro, 2005).

and the Catalan Coastal Cordillera (Gava, Moncada) in Barcelona. For the studies conducted so far, it seems that the only deposits of variscite and turquoise exploited in Prehistoric Europe were in the Iberian Peninsula. Mines of prehistoric turquoise and variscite at Gavá (Barcelona) are well known, although there are clear indications already confirmed of prehistoric exploitation in Encinasola (Huelva) and Palazuelos de las Cuevas (Zamora), where exploitation is manifest in Roman times *(Campano Rodriguez & Sanz 1985; Domínguez-Bella 2004)*.

Variscite mining of Catalonia (Gavá mines) seems that supplied the northern and northeastern Spain and southern and western France *(Guerra et al. 1995; Edo et al. 1998)*. The deposits of western Spain seem that variscites distributed to the southwest and western half of the Iberian Peninsula *(Domínguez-Bella 2004)*, although there is evidence of a large distribution networks over long distances, variscites have been identified from these sources in the French Brittany *(Querré et al. 2012) (Fig. 6)*.

Other variscite deposits of the Iberian Peninsula that has certainly been exploited since prehistoric times are Encinasola (Huelva) and the area of Palazuelos-The Bostal-Bragança *(Moro et al. 1992 a-b; Moro et al. 1995 a-b; Domínguez-Bella 2004*) located in the SW and NW of the Iberian Peninsula, which have been analyzed in the last 18 years *(Dominguez-Bella & Morata 1995; Merielles et al. 1989; Domínguez-Bella 2004 ; Domínguez-Bella et al. 2004; Odriozola et al. 2010; Querré et al. 2012)*. We have made in recent years, different geochemical analysis along with statistical studies by factor analysis of analytical data obtained by ICP-MS-LA, XRF and SEM-EDX, with a geochemical model of 13 variables, carried out both on geological samples and archaeological samples *(Dominguez Bella et al. 2002)*. Recently, further analysis by PIXE-PIGE, XRF, PXRF, XRD are being carried out on these geological sites of the Iberian Peninsula, including minor ones like Punta Montalvo, Coruña and several archaeological sites in Spain, Portugal and France, in an International Project, CALLAIS, in the CHARISMA program of the EU 7th Framework. We also propose new isotopic studies for these variscite materials as a complementary line of future research in the determination of source areas.

In the case of turquoise, very different analytical techniques have been used for mineralogical characterization and source areas identification. Techniques as Arc emission spectrometry analysis, Electron microprobe, Instrumental neutron activation analysis, Spectrometry, X-ray diffraction and X-ray fluorescence are employed throughout the world. As an example, we can cite the study on a set of cylindrical beads and zoomorphic of Tafí Culture (300 B.C. - 800 A.D.), Tucuman, Northern Argentina, possibly from the turquoise deposits in northern Chile *(Fig. 7) (Domínguez-Bella & Sampietro 2005)*. The same method as the variscites has been

Fig. 8. Smoky rock crystal, monocristaline quartz from the Dolmen de Alberite I, Villamartín, Cádiz, SW Spain. (Domínguez-Bella & Morata, 1995).

employed in the Iberian turquoise, since it has the same paragenesis in the studied deposits.

5.1.2. Single crystal quartz

The presence of quartz crystals is relatively common in Prehistoric funerary environments, in the southwest peninsular, in the province of Cadiz, as in many other parts of the Iberian Peninsula and Europe. Spectacular examples are the large single crystal of smoky quartz that appeared in the dolmen of Alberite *(Fig. 8)*, a rock crystal in the dolmen of El Juncal, Cadiz or a quartz crystal of Triassic age, as found in the silos of La Esparragosa archaeological site (Chiclana).
The first is a pegmatitic quartz, accompanied by small traces of feldspar. The possible source area of that can be located, according to *Dominguez-Bella and Morata (1995)* in pegmatitic rocks environments, perhaps in the Sistema Central of Spain, with which pegmatite quartz crystals have strong similarities in their morphology, and located several hundreds of kilometers away where it appeared.

In the case of the bipiramidal quartz crystal appeared in La Esparragosa archaeological site (Chiclana), it is a crystal type "*Jacinto de Compostela*", grey in color and a size of about 3 cm. These crystals are common in gypsum and clay deposits of the Keuper facies, of Triassic age in the Betic Cordilleras. Outcrops of these materials extending along a band from southwest to northeast across the province of Cadiz, according to the predominant direction of the Betic Cordillera, several of them exist in this area of the Atlantic band of Cádiz, in Iro River Basin in where the site of La Esparragosa is placed. Thus, the origin of this crystal would probably be local, being of an exceptional size, it was possibly collected.

5.1.3 Amber in the Prehistory of the Iberian Peninsula and Europe

In addition to many of the minerals and gems known and used since ancient times, we could include in this group of substances other compounds of organic origin as fossil resins, which also have been used in jewellery or as objects of prestige, from prehistory to present.

From the Upper Palaeolithic, pieces of amber are recorded at sites attributed to the Magdalenian in locations of Central Europe, also in enclaves of the Hamburgian culture. The Neolithic records are also very prominent and many beads are well known since ancient in the Megalithism of the Iberian Peninsula, especially dolmens in Portugal. They have generally been classified as jet, due to the total absence of analysis.

The ambers of Baltic origin are composed mainly by succinite *(Beck et al. 1965; Stout et al. 2000)* and are used in Europe since at least the Iron Age, with a widespread use during the Roman Empire, at both the European and Mediterranean areas, where amber routes were well established, crossing Europe from north to south. This characterization has been worked since the 70's *(Savkevich & Shaks 1964; Beck & Vilaça 1995; Kosmowska-Ceranowicz 1990, 1999 & 2003)* on geological and archaeological ambers, especially in European sites.

For organic compounds such as amber, the fundamental techniques that have been used since the beginning of the analytical archaeometric have been FTIR *(Beck et al. 1964)* and some other partially destructive as elemental analysis *(Domínguez-Bella et al. 2001)*. Considering the great experience on the topic of the schools in Northern and Eastern Europe, it follows that one of the best methods of identification and classification of resinites is infrared spectroscopy (FTIR) *(Savkevich & Shaks 1964; Beck et al. 1965)*.

Angelini & Bellintani (2005) published a study about five different localities and types of European geological ambers, and also included Italian geological ambers from seven different deposits. They emplo-

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Fig. 9. Neolithic necklace of amber and variscite from the Chousa Nova dolmen, Galicia, NW Spain (reconstruction). (see Domínguez-Bella & Bóveda, 2011).

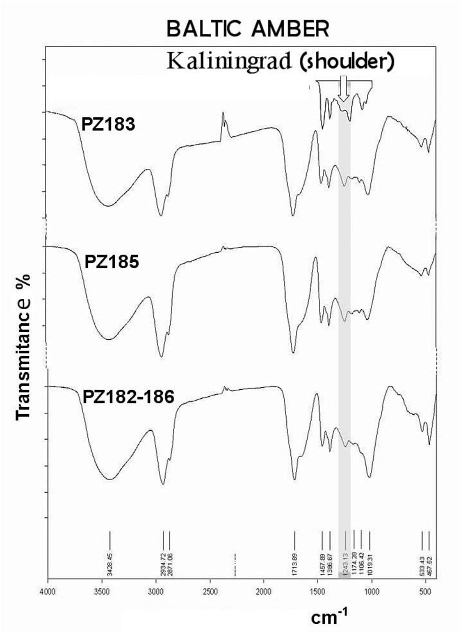

Fig. 10. FTIR diagram from three archaeological amber beads of the necklace from Chousa Nova dolmen, Galicia, NW Spain, and comparison with a succinite Baltic amber sample. (Domínguez-Bella & Bóveda, 2011, modified).

yed in the analysis Fourier transform infrared spectroscopy (FTIR) and diffuse-reflectance infrared Fourier transform (DRIFT).

Other techniques of analysis of geological and archaeological ambers are the GC-MS and thermal pyrolysis analysis, X-ray diffraction and scanning electron microscopy and mass liquid chromatography with spectrometric detection. In some cases, the use of polarizing transmitted light microscope in the study of natural polymers in thin sections is another important technique for the identification of the mineral inclusions in the sample, the textural relationships between them, the Investigations of the fluid inclusions and the fossil animals and plants fragments. In order to obtain a fingerprint related to the origin amber, good results are obtained by direct mass spectrometric techniques, as the atmospheric pressure photoionization (APPI) *(Tonidandel et al. 2008)*. The results using the X-ray diffraction virtually does not allow in general, to obtain information of interest *(Domínguez-Bella et al. 2001)*. However there are cases where it provides information about the type of amber inclusions or minerals, such as occurs with the romanite *(Teodor et al. 2009)*.

These new techniques for this cataloguing of deposits and varieties of ambers and the creation of analytical databases of the same, will undoubtedly improve the determination of the origin of archaeological ambers. This is especially important to identify the source areas of different ambers of local origin, which are different chemically and genetically in relation to the Baltic succinite *(Teodor et al. 2009; Kosmowska-Ceranowicz 1999).*

These studies have been generally associated with the search of the geological source sites in each region. In the Iberian Peninsula, some ambers have been characterized in different archaeological sites; in southern peninsular *(Dominguez-Bella & Morata 1995)*, Portugal *(Vilaça et al. 2002)*, north of Spain *(Alvarez et al. 2005; Peñalver et al. 2007)*, central Spain *(Domínguez-Bella 2010)* and Galicia *(Domínguez-Bella and Bóveda 2011)* (Fig. 9).

In the case of amber there are several records in the area of the province of Cadiz, SW Spain, with necklace beads made of amber and in a Neolithic chronological context, such as the Dolmen Alberite I, whose origin seems to be far off, since the samples corresponds to a simetite, species unknown in geological outcrops of the Iberian Peninsula, at least until the present *(Domínguez-Bella et al. 2001)*. We are currently working on Galicia archaeological ambers *(Domínguez-Bella & Bóveda 2011) (Fig. 10)* and on two new Neolithic sites in this region of southern Spain, having identified new ambers beads or pendants without Baltic provenance.

6. Conclusions

The interest from the fields of archeology and heritage management and study of the mineralogy and archaeometry is growing in recent decades in a very strong worldwide, with an increasing number of scientific publications and outreach related to the archaeometric application of techniques, where the mineralogy plays an important role in the study and conservation of heritage.

We agree with the idea of mineralogists colleagues in the need for a real interdisciplinary work in the archaeomineralogical and archaeometric determinations which we have been doing in our research field in recent decades.

There are a great number of mineralogical analysis techniques currently available, the choice of which one or ones are best suited to each case studies posed problems and it is an important matter to determine a priori. It is usually necessary more than an analytical technique for a complete study of the samples and the resolution of the issues raised.

Undoubtedly, the physical availability of equipment and the economic cost of the use of certain analytical techniques will largely condition their use in solving archaeometric problems, the number of samples to be analyzed, the existence of scientific equipment in the surroundings and the available budget. Access to large facilities has been provided in recent years, at least at European level, with programs such as CHARISMA, the 7th Framework Program for transnational access to large equipment.

The development of analytical databases for different substances such as amber, ancient metallurgical products, marble, flint, ceramic, etc. undoubtedly facilitate future research in the field of archaeometry, but require large investments or transnational projects which must overcome many administrative difficulties, economic and technical to be of homologated use. The different analytical techniques and technological developments, and experimental protocols make it difficult to homogenize the results contained in these bases, at least for specific techniques. Databases of images of OM, CL, in research areas such as marbles and rocks or ceramics in ancient times are for example very interesting.

A massive growth has been experienced in the Iberian Peninsula in recent years regarding the number of researchers involved in archaeometry work, although there is still a long way off, given the historical gap in relation to other neighbouring countries. The increasing international collaborations are softening rapidly these differences.

From our own experience we can note that the information provided by mineralogical techniques applied to archaeometry is providing valuable data for historical reconstruction in different periods of the Prehistory Peninsular, European and North African. This is applicable to the aspects related to daily use objects in stone, ceramic, bone, metal, etc., as to others related to rituals, symbolism and power, where minerals like pigments and minerals, rocks and substances used in jewelry and votive or prestige objects would be taken into consideration.

We have not only been able to characterize geochemically many compounds mineralogical and minerals, rocks, ceramics and pigments, but we have also obtained interesting information about the source areas of these materials and hence their mobility at short, medium or long distance, during prehistory. Notable examples can be used as a pigment cinnabar in many megalithic tombs of the Neolithic and the

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Chalcolithic, in which the determinations mineralogical, geochemical and especially the S isotopes seem promising lines of work. Amber, with a presence since the Paleolithic in caves of the North Spain, has been used as a jewel in the Neolithic-Chalcolithic of Galicia, Castile and Andalusia, with a possibly peninsular origin, while from the Iron Age, is becoming a priority Baltic provenance.

Tools or prestige objects manufactured in polished rocks have also provided valuable archaeological information, after determining their local or distant origin, possibly transported over long and organized exchange networks during recent prehistory. So it is with some knapping stone products, especially in siliceous rocks as flint and radiolarite, where objects such as large blades of flint, are transported hundreds of kilometers along the peninsula and Europe. Another of the minerals used in the manufacture of polished stones, used as tools or objects of prestige are the sillimanite axes and adzes, with great peninsular diffusion which extends to the West European and even the British Isles, with relatively few areas-source and in which analytics we are still immersed.

Materials used as jewels of prestige as variscite and turquoise, with geological source areas quite well known and almost exclusive to the Iberian Peninsula, show large geographical mobility, exceeding of a thousand kilometers as occurs with jadeite and other HP alpine rocks in their distribution network throughout Western Europe. In this case, mineralogy, geochemistry of trace elements and multivariate statistical treatment of analytical data, are allowing their potentially-source geological areas.

These are undoubtedly exciting topics and with great future expectations in the scientific development of archaeometry and mineralogy, which certainly are of great importance in understanding the complex human history and for some mineralogists, continue to constitute a "fatal attraction".

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