



# Book of abstracts

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**X-1. Archaeometry of prehistoric  
and protohistoric stone, metal,  
ceramics and glass**

# Investigation on Neolithic adornments using X-ray spectrometry

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Adornments from some Neolithic sites (Boian and Gumelnitza Kocadermen-Karanovo VI cultures) situated in South of Romania have been investigated to determine their mineralogical composition using X-Ray Fluorescence (XRF) and Scanning Electron Microscope Energy Dispersive X-ray Spectroscopy (SEM-EDS). Pendants and beads were preliminary analyzed with a XMET 3000TXR+ Oxford Instruments XRF portable spectrometer (elements from Potassium to Uranium) and, for light elements (Carbon, Oxygen, Sodium, Magnesium, Aluminum, Silicon) which are essentially for minerals characterization, investigated using a Zeiss EVO MA15 electron microscope. The most spectacular results are the identification of two pendants (ear-rings?) from nephrite, of few beads from native copper embedded in malachite and of a necklace from malachite beads put together with different types of nephrite beads. A comparative analysis with similar artefacts from Serbia (e.g. Antonovic and Stojanovic, 2009, The nephrite amulet from Zmajevac) and Bulgaria (e.g. Kostov, 2010, Gem minerals and materials from the Neolithic and Chalcolithic periods in Bulgaria and their impact on the history of gemology) is presented. Some considerations about geological provenance (deposits) of nephrite, jadeite and native copper are also discussed.

**Keywords:** adornments, Neolithic, X, ray spectrometry, nephrite, malachite, copper

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\*Speaker

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# Ceramic pigment analyses: Archaeometry to understand its use.

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The archaeological site RS-TQ-141 is located in the municipality of Cruzeiro do Sul, Rio Grande do Sul (Brazil) and it Guarani Tradition. The site is located between a plantation area and a large slope that extends to the bank of the Taquari River. The site comprises an assemblage of 642 ceramic fragments, 88 lithic objects and 17 samples of coal, soil and bone material. Of these, about 200 small fragments of ceramic present internal and / or external painted surfaces; however, there are issues concerning the shape and size of the vessel. Moreover, it is problematic in determining being the form of the vessel (e.g. pot, bowl, storage jars or drinking vessel). In the past, there have been several studies that have considered the functionality of these vessels, however, little progress has been made in terms of the analysis of the painted surfaces, be it its function, meanings or pertinent to our paper, the chemical analysis involved in the composition of pigments. Applying  $\mu$ -FTIR methods it was possible to differentiate white and red pigments produced with different types of (ocher) clays. The pigments were produced with the same groups of clays used for the manufacture of ceramics. The presence of at least two groups of clay is recorded: one with more calcium (montmorillonite) than the other, and the other with more aluminum (kaolin). The white pigments are distinguished in the spectra by the presence of carbonate and more aluminum, and by the absence of magnetite. From this research, there does not seem to be any difference between internal and external pigments. Organic materials were detected, both in the pigment and in the production of the ceramics; the latter probably is related to pyrolysis phenomena. The organic residues present within the analysis could suggest the ceramic was used to cook foodstuffs and these in turn became attached to the pigments.

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**Keywords:** FTIR, Pottery, Pigment, Clay, Organic



# A multi-methodological characterisation of flint: a case study from NW Belgium

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In most regions of Europe, flint was by far the most important raw material for the production of stone tools during the Stone Age. The characteristics of flint material used for stone tool production in NW Belgium are investigated in this study. Due to its formation process, flint is defined by a wide variety of internal structures, chemical variations and impurities. Moreover, flint weathering and the resulting alterations make the study of this material even more complex. Flint weathering is often expressed as patination, which is linked to the geological and archaeological context, and mostly present on Palaeolithic artefacts. Furthermore, burned artefacts appear throughout the entire Stone Age as a result of intentional (heat-treatment) or unintentional heating (forest or camp fires). The preservation of flint artefacts is influenced by these weathering processes, which are important aspects to consider in use-wear analysis as use-wear traces can be partially or completely obliterated. For this reason, altered artefacts are often neglected during analysis and may bias our understanding of the use of stone tools by prehistoric man. Therefore, it is important to understand how the flint characteristics influence weathering, and what the impact is of weathering on the preservation of use-wear traces. The first goal of this study is investigating the characteristics of flint material using a combination of traditional techniques such as optical microscopy, X-ray fluorescence (XRF) and scanning electron microscopy (SEM). Additionally, the possibilities of high-resolution X-ray computed tomography (micro-CT), providing 3D information of internal structures of flint, are explored because of its non-destructive nature and its potential for future time-lapse weathering experiments. The combination of above mentioned techniques allows us to work out a characterization protocol at macro- and microscopic scale, combining mineralogical, chemical and structural information. This protocol adds a new perspective to the worldwide ongoing research on flint characterisation and its archaeological significance. The selection of the raw materials is based on lithic finds excavated at various prehistoric sites along the Scheldt river, assumed to be made of raw material found in these areas. Therefore, the raw materials selected for this study are all located in the western part of Belgium (late Cretaceous outcrops near Mons, Lille and Tournai) and close to the border with the Netherlands (Western Scheldt, Vlissingen).

**Keywords:** flint, characterization, weathering, micro CT, optical microscopy, geochemistry

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# Loess is more: chemical and petrographic analyses of Middle-Late Bronze Age ceramics and reference clays from the Great Hungarian Plain

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In much of Europe, the Bronze Age (~3000-1200/500 BC) witnessed dramatic increases in political hierarchy and wealth inequality. Control of resources and trade routes was likely a driving force behind these social, political, and economic changes. The K'or'os region of the Great Hungarian Plain (GHP) retains relatively egalitarian burial patterns, little evidence for accumulation of personal wealth, and a dispersed pattern of metal production suggesting that the region did not participate in these broader trends. In the absence of large volumes of longer-distance imports, it is however difficult to assess how the region articulated with neighboring areas and broader Bronze Age networks. We present the results of petrographic and geochemical analyses of ceramics from late-Middle Bronze/early-Late Bronze Age (~1600-1300 BC) sites on the Great Hungarian Plain, primarily focused on the cemetery of Békés 103, excavated by the BAKOTA (Bronze Age K'or'os Off-Tell Archaeology) project. Comparison to an extensive sample of reference clays taken from across the GHP shows that despite the relative geological uniformity of the GHP, subtle compositional differences exist in clays and ceramics that can be used to assign sherds to general production regions, and that imports can be distinguished at individual sites. We discuss these results in the context of stylistic patterning in ceramics at Békés 103 as well as broader patterns of trade and exchange across the region.

**Keywords:** Ceramic, Geochemistry, Petrography, Hungary, Bronze Age

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# Petrographically similar but chemically different: the application of LA-ICP-MS on the mineral inclusions in the sourcing of ceramics raw materials

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The analysis of archaeological ceramics can be realised from multiple methods: the petrographic analysis with polarising microscope on thin sections, by X-ray diffraction (XRD), the global chemical analysis of paste generally by spectrometric methods (XRF, INAA, ICP-OES...). However, in some cases, these analyses do not allow us to determine the geographical origin of the raw material used to shape the potteries. It is the case for pots mounted with clays coming from the weathering of granitic and granito-gneissic rocks (eg. granites, gneiss, gabbros...), because of the similarity of the mineral inclusions assemblies present in the pastes of the ceramics. Indeed, the lack of distinctive features prevents accurate identification of the existence of different petrographic groups. Furthermore, the global chemical compositions of this type of ceramic may vary due to the presence of larger numbers of certain mineral species or large differences in the size of inclusions. It was therefore necessary to develop a new approach to distinguish the sources of raw materials used by potters. Therefore, we have developed a method based on the analysis by Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) of mineral inclusions (eg. biotite, opaque minerals, amphibole) present in ceramic pastes. We compare the chemical composition of the mineral with the same mineral from the supposed mother rock and, as a result, discriminate different raw materials sources. In this communication, we will present our research and development we have made in the field of ceramic sourcing, through several case studies distributed over a large period (from the Neolithic to the Late Iron Age), located on different islands of Brittany in western France.

**Keywords:** Ceramics, raw materials, sourcing, LA, ICP, MS

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\*Speaker

# The function of early ceramic container and subsistence strategies in Zamostje 2, Central Russia, during the early Holocene

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In north-eastern Europe, resource-rich aquatic and boreal ecotopes were created with the stabilization of climate during the early Holocene, with a climatic optimum from ca. 8ka cal BP. During this period, pottery technology also dispersed across the continent and was taken up by a broad range of hunter-gatherer societies. We aim to explore how early pottery-producing hunter-gatherers adapted to these new conditions and the relationship between pottery and their subsistence economy. This study focuses on the site of Zamostje 2, located 110 km north of Moscow in Russia, along the Dubna River, one of the most important sites in this region due to its remarkably preserved, uninterrupted stratigraphic sequence from Mesolithic to Middle Neolithic (Lozovski and Chaix, 1996). The site was occupied during the Atlantic period from around 7,000 to 5,500 cal BC. The site has produced a very significant collection of well-preserved artefacts and ecofacts. Faunal remains at Zamostje 2 site suggest a broad subsistence economy based on hunting/gathering/fishing throughout the late Mesolithic and Neolithic (Losovski and al. 2013), the latter period defined by the introduction of pottery. In order to examine the motivation for its introduction, we aimed to test whether pottery had a specific function or alternatively were used for processing a broad range of foodstuffs. To do this, we undertook molecular and isotope analysis of lipids extracted from 135 samples of absorbed and superficial organic residues on ceramics from Zamostje 2, using GC/MS and GC-c-IRMS. The results are compared to the use of other food-processing technologies (lithic, wooden artefacts, basketry) which are exceptionally preserved at this site, and to the botanical and faunal records.

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\*Speaker

**Keywords:** Ceramic, organic residues, lipid, isotope, Russia, Neolithic

# New data on the protohistoric exploitation of obsidian in Nakhchivan: first provenance results from the PAST-OBS project

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Started in 2016, the PAST-OBS project ‘*From transhumance to the mine: the role of nomadic pastoralists in the protohistoric exploitation of obsidian in Iran and the Caucasus*’ (dir. Dr. Le Bourdonnec; LaScArBx ANR-10-LABX-52) aims to investigate the exploitation of natural resources and the rise of mobile pastoralism in the Lesser Caucasus from the Neolithic to the Early Bronze Age (ca. 6200-2200 BCE). This project is connected to the French-Azerbaijani ‘*Middle-Araxes Archaeological Project*’ initiated in 2006 (dir. Dr. Marro) and focuses on the reconstruction of the complex relationships between mobile pastoralists, the exploitation of natural resources, and technological innovations in northern Iran and Azerbaijan, primarily through the comprehensive study of the obsidian assemblages unearthed from a series of archaeological sites in Nakhchivan (Azerbaijan) dated from the Neolithic to the Bronze Age. So far, it includes the excavated settlements of K’ul’tepe I, Nakhchivan Tepe, Ovçular Tepesi, Uçan Agil, and Sorşu, as well as the surveyed sites of B’ul’ov Qayasi, Çay Ağzi, Kolani, and Mesmeliagil. To allow, in a timely manner, the representative and non-destructive geochemical characterisation of the large assemblages – which altogether amount to several thousands of artefacts – a flexible analytical strategy relying on complementary analytical methods was developed, mostly using portable X-Ray Fluorescence spectroscopy [pXRF] and Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry [LA-ICP-MS]. Together with the information provided by the typo-technological study, the results obtained following this strategy helped us gain a significant insight into the complexity and diversity of the obsidian consumption behaviours that developed in the Lesser Caucasus from the Neolithic to the Bronze Age.

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**Keywords:** Caucasus, Nakhchivan, Protohistory, Obsidian provenance, Nomadic pastoralism

# Amber, beads and social interaction in the Late Prehistory of the Iberian Peninsula. An update

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The identification of archaeological amber has been used in Iberian prehistory to evidence long-distance exchanges and engage Iberia in networks that connect western Europe with central and northern Europe, the emergence of social complexity, and the consolidation of trade networks. However, until now, no comprehensive analytical study of the Iberian amber has been produced to support any of the interpretive models currently in use.

This paper approaches the analysis of Iberian Peninsula amber artefacts by considering their provenance (based on FTIR characterization), chronology, and spatial relationship with other exotica. Our work increases the number of analyzed artefacts to 156 (24%), out of the c. 647 currently known for the Iberian Peninsula.

Based on these new data and a review of Murillo-Barroso and Martín-Torres (2012), this overview outlines amber consumption patterns from the 6th to 2nd millennia BCE and demonstrates long-distance amber exchange connecting Iberia with the Mediterranean region from the Neolithic period onwards.

**Keywords:** amber, FTIR, Iberia, provenance, Prehistory

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\*Speaker



# The fingerprinting of roman gold by LA-ICP-MS: the AUREUS project

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This paper aims to present the methodological approach developed by LA-ICP-MS that is used in the AUREUS program (2016 –2018), for the fingerprinting of gold by the analysis of gold coinage. The AUREUS project directed by Pr. Arnaud Suspène, links numismatists and analysts in the aim to understand the establishment of the first unitary gold coinage of occidental Europe: the Roman gold coinage. This multidisciplinary approach allows to establish the evolution of the gold stocks used for coinages between the end of the IVth century BC and the Ist century AD. At the end of the study almost 1000 coins from the main gold coinage areas (Africa, Greece, Italy, Celtic world, Spain) and pieces of jewellery from French patrimonial collections will have been analysed to determine the links between gold used by artisans, through their chemical fingerprints. The method used is based on several micro sampling by DPA-LA-HR-ICP-MS (Depth Profile Analysis by Laser Ablation coupled to a High Resolution Plasma Mass Spectrometer) at the IRAMAT-CEB laboratory (UMR 5060, IRAMAT-CEB, CNRS/university of Orléans). In this way, it is possible to determine the variations of the concentrations of the major, minor and trace elements from the surface of the coin to a depth ranging from tens to several hundred micrometres. Thus, this method allows us to avoid issues due to surface enrichments owing to the alteration of the metal through time but also to identify voluntary gilding certain flans sometimes carried out by the artisans in official mints to disguise low gold contents. The elemental analysis of gold coins makes it possible to understand the monetary policies by the different authorities. Moreover, thanks to the concentrations of platinum and palladium that are reliable elements to trace gold, it is possible to link the gold raw material employed for coin production between civilisations and determine the evolution of the origin of the supply and exchanges through time.

**Keywords:** Gold, raw materials, LA ICP MS, Antiquity, Coinage

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\*Speaker

# Microstructural analysis of pipe-shaped iron oxide used as red pigment in prehistoric art of Japan

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In this study, the internal microstructure of pipe-shaped red iron oxide used as a coloring material of cave painting and pottery in the prehistoric period of Japan was confirmed with the aim to elucidate the origin, and the mechanism of its formation and color development. Pipe-shaped iron oxide particles excavated from the moving remains of the Chiba Prefecture, Ichihara in Japan (Middle to Late Yayoi period, provided by Mr. T. Kamijo, Figs. 1 and 2), have been investigated using optical microscope, FIB-SEM, and TEM.

Based on the optical microscopy and SEM observations, the pipe-shaped red iron oxide particles have lengths of 5–20  $\mu\text{m}$ , widths of 1–1.5  $\mu\text{m}$ , and wall thicknesses of 200–300 nm, with large dispersion in size; each pipe itself comprises an aggregate of fine particles of approximately 100 nm in size. SEM-EDS analysis revealed that, in addition to Fe, trace amounts of Si, P, Ca, Al, Mn, S, and Ti are present in the pipe-shaped iron oxide particles. Based on the results of the TEM observation, a structure comprising three phases, namely, a region consisting of iron oxide, a region consisting of Si, and particles containing large amounts of P and Ca (calcium phosphate), was found. Al was also found to be distributed throughout the particle. Because the presence of calcium phosphate in the pipe-shaped iron oxide particles was confirmed, there is high probability that they originated from iron-oxidizing bacteria. Therefore, in the future, we intend to verify the distribution of calcium phosphate in the pipe-shaped red iron-oxide particles while continuing the identification of the type of calcium phosphate and the state of iron oxides. This study is the first case examining the internal microstructure of pipe-shaped red iron oxide particles. We are striving to gain further insights via detailed analysis of the results acquired in the forthcoming work while promoting cooperation among experts of related fields, such as archeology, biology, and geology.

**Keywords:** Red pigment, pipe, shaped, iron oxide

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\*Speaker

# Multianalytical characterisation of Egyptian Flint as a means for understanding seasonality in the Nile valley and Western Desert (Egypt)

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Seasonal mobility of ancient populations occurred during Early Holocene in the area of the Second cataract of the Nile River (Northern Sudan) and the Western Desert region (Southern Egypt), as attested by various different archaeological indicators, among them pottery and lithics. A distinct feature in the lithic assemblages is the presence of a flint type that has been called "Egyptian flint".. The main macroscopic characteristic of the Egyptian flint is its dark and pale grey colour. The particular cherts association crops out mainly in the Western Desert in the Eocene limestones and it is not present in the geological substrate around the second cataract area. However, along the Nile valley, it is possible to find chert-cobbles in the fluvial secondary deposits, probably deriving from other chert-rich deposits of Jurassic age, cropping out in the area of Jebel Abyad (West of Dongola, Sudan, at the latitude of the third cataract). It is not clear if there is a common chert-source for both lithic assemblages found at the second Cataract and in the Western desert sites was used, implying the trades of these objects, or if the flint was worked and used locally. Therefore, in order to determine whether the dark grey/grey Egyptian flint from the second Cataract and Western Desert derive from the same source, a microstructural, compositional and mineralogical analysis was performed on the samples.

For this purpose we analysed a set of 22 flint tools from 11 sites of the Western Desert region (Egypt) and near the second Cataract (Sudan), sampled from two lithic collections (Wendorf and Colorado) stored at the British Museum (London, UK). SEM, mXRD, LA-ICP-MS and mPIXE analyses were performed to characterize the considered tools. The microstructure of the flints, observed at the SEM, resulted to be in most of the cases not homogeneous, composed mainly by microcrystalline quartz, associated to numerous microfossil traces, dolomite and calcite crystals, apatite fragments and minor oxides, sulphides and sulphates, in few cases with crystal dissolution traces and pores.

Mineralogical analysis indicates that in addition to the quartz, these samples contains also

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small amount of moganite, mainly crystallised inside microfossils, as attested by micro-Raman mapping.

The moganite is accompanied by higher Fecontent than the surrounding microcrystalline quartz, as observed from mPIXE mapping and LA-ICP-MS analysis: this element seems to favour the moganite crystallization.

**Keywords:** Sudan, Nubia, Western Desert, Egyptian Flint, Early Holocene, seasonality

# Non-Destructive Elemental Analysis of 20,000 Stone, Metal, Ceramic and Other Artifacts by pXRF: Issues on Surface Analyses, Sample Heterogeneity, Detection Limits, and Calibration

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A portable, hand-held X-ray fluorescence (pXRF) spectrometer has been used for more than a decade to measure the major and/or trace element composition of more than 20,000 stone, metal, ceramic and other artifacts. Different brands and models have been used, analytical settings have changed, and calibration has been developed for some materials. Issues have been raised about analyzing materials like ceramics which may be heterogeneous, with temper added to the clay, as well as slip or paint on the surface. In addition, the surface of many artifacts (e.g. corroded metals) may not represent the bulk composition, since they may be degraded and/or contaminated over time. All analytical instruments have detection limits and varying precision of its measurements, while X-ray analysis methods need material-specific calibration due to matrix effects. Unfortunately, many archaeological applications of pXRF have been performed without full consideration of these principles, and discussion of analytical limitations in subsequent publications.

In this presentation, I show the clear success of using pXRF for obsidian analyses in Europe and the Mediterranean - a fairly homogenous material from a limited number of geological sources and little if any change due to production, use, and being in the ground for thousands of years. The ability to conduct non-destructive analyses on thousands of artifacts within many different museums and storage facilities has resulted in statistically significant patterns of distribution and trade over 4500 years.

Metal objects may be heterogeneous, affecting studies using any analytical method, and often are significantly corroded, while museums frequently limit destructive analysis of objects on display. This is partly resolved by testing multiple spots and at least semi-quantitatively measure the copper, arsenic, tin, lead, iron and other elements in alloys, while addressing patterns and changes in technology over time, as well as identifying recycled objects, mislabelled artifacts, and forgeries.

Ceramic analytical studies address where the clay originated and the organization and scale of production of both everyday and fine wares. Analysis of unpainted/unslipped surfaces or

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edges avoids surface issues, but pXRF analyses do not produce results for as many trace elements as do INAA and LA-ICP-MS. Depending on the research project, this may limit how specific clay-source interpretations may be made.

Along with analyses conducted on other materials, including marble, bones, and soils, it is necessary for the user to understand the limitations of both non-destructive analyses, the particular instrument (i.e. pXRF) involved and discuss that clearly in any resulting publications.

**Keywords:** pXRF, obsidian, elemental analysis

# Methodological approach in ochre provenance studies: preliminary results on the so called Cheops' ochre from the Western desert of Egypt

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The scientific approach in ochre provenance studies is generally based on the use of geochemical analysis (Green and Watling, 2007; Popelka-Filcoff *et al.*, 2008; Sunday Eiselt *et al.*, 2011; Bu and Cizdziel, 2013; MacDonald *et al.*, 2013) even though recently the adoption of microscopic (PLM and SEM/EDXS) and mineralogical analysis (XRPD) (Pradeau *et al.*, 2016; Salomon *et al.*, 2008; Dayet *et al.*, 2016; Cavallo *et al.*, 2017a; 2017b; 2017c) demonstrated their validity and suitability in such research.

The possibility to study the raw materials collected during an expedition carried out many years ago in the Southern Egyptian Great Sand Sea desert, gave us the opportunity to adopt the same methodological approach with the aim to create homogeneous groups of sample on the basis of mineralogical, textural and micro-structural features. Yellow and red ochre samples are associated with the Aptian Abu Ballas Formation which represents a thin marine intercalation within the continental sandstones of the Nubia Group (B'ottcher, 1985). Preliminary XRPD analysis allowed to state that the mineralogical composition of the analysed samples is very similar. They are composed of kaolinite, quartz and hematite or goethite depending on the colour.

This research represents a unique attempt to focus on raw materials which were presumably used in prehistoric settlements in the outcropping area but also during the Dynastic age (Negro *et al.*, 2005).

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**Keywords:** archaeometry, ochre, provenance, prehistoric settlements, Egypt

# Archaeometry of fire aided limnosilicite mining in the Avas-T'uzk'oves (Miskolc, NE-Hungary) paleolithic silica source

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Our experiments and analytical investigations were done on silica rock outcrops from Avas-hill (Miskolc, centre of Older Stone Age research in NE-Hungary), where the excavations run by Árpád Ringer during 1988-2005 revealed mining sites, exploited by the help of fire. The geological formations of limnosilicite are layered limnic silica beds precipitated from cold water solutions. A large number of tools were discovered in NE-Hungary and SE-Slovakia, with their source tracked back to Avas-T'uzk'oves. The tools and associated technological debris usually display signs of alteration by heat – thermal stigmas – associated with characteristic hematite red colour. However, similar colours could be developed during the silica precipitation also, making the interpretation biased. In search for more concrete evidence for thermal stigmas, we turned to mineralogical investigations of experimentally heated raw silica from the mining source. Our results revealed, that a very simple mineralogy is characteristic for the Avas-T'uzk'oves limnosilicite: dominantly nanocrystalline quartz, subordinated moganite and amorphous silica. Minor limonite and goethite is present, creating some brown banded-striated textures. Chemical analysis also supports the high purity silica composition of the material. Upon direct fire-setting on a simulated mining site and laboratory oven heating, we determined that red colour is given by limonite and goethite oxidation above 250 °C. At 370 °C the large blocks were instantaneously fragmented into blades and shards. Continuing mineralogical investigations on samples heated to 250 °C and 350 °C and applying Fourier-transformed infrared spectrometry (FTIR), we have evidenced that molecular H<sub>2</sub>O is present in the material, which is removed by heating. Microinclusion of H<sub>2</sub>O were trapped during precipitation and diagenesis in the fibrous limnosilicite texture. By heating, they develop pressures high enough to fragment the silica texture starting from 360 °C, associated with a crystallization of quartz from amorphous silica and recrystallization of nanocrystalline quartz. FTIR analysis show convincing evidence for both H<sub>2</sub>O loss and quartz thermal evolution, being a suitable decision making method for fire aided mining of limnosilicites similar to the Avas-T'uzk'oves material.

**Keywords:** limnosilicite, fire, setting, ATR, FTIR, thermal stigma

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\*Speaker

# Chemical characterization via pXRF of Bronze Age metal items from the National Museum of Archaeology of Valletta, Malta

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We have performed non-destructive analyses on 21 metal artifacts from the Maltese archipelago and dated to between the 17th and the 12th century BC, representing the only evidence of metalwork in that country during the Bronze Age. The objects - daggers, axes, metal vessels, rings, pins, generic tools and an ingot - were found in the key sites of the local Early Bronze Age (Tarxien Cemetery) and Middle/Late Bronze Age (Ghar Mirdum, Borg in-Nadur, Bahrija) and were traditionally interpreted as made out of bronze solely on the account of a direct exam. This evidence was considered by scholars as the indicator of commercial relations between the people of Malta and Mycenaean entrepreneurs active in the Mediterranean and responsible for the introduction of pottery, metals and other exotic goods in nearby Sicily. The presence of supposedly bronze items in Malta and Sicily, both with geographic contexts lacking in raw metals, was traditionally interpreted either as the result of a consistent flux of Mycenaean imports, with a climax during the Late Helladic IIIA-B period, or as the outcome of a significant contribution of Mycenaean visitors to the development of local metallurgy. However, the recent results of our pXRF analyses (portable X-ray fluorescence spectrometer) on metal artifacts from Bronze Age Sicily have demonstrated that the majority of the samples are made of copper and not of bronze, and therefore it is unlikely that they are Mycenaean imports, shifting to a later date the beginning of metal alloy working in Sicily. The aim of this contribution is to present the results of a research carried out on the Maltese samples applying the same technique, in order to ascertain the chemical composition of these 21 artifacts, to establish the origin of the metals implied by their composition, and to compare the data with those already available for Sicily and the Aegean. The interpretation of the results certainly contribute to the reconsideration of the impact of Mycenaean culture on the development of the Maltese Bronze Age culture, eventually clarifying the role of Sicily in Maltese-Aegean interactions.

**Keywords:** Bronze Age, Metallurgy, Malta, Mycenaean Trade, pXRF

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# Variscite provenance analysis. A review

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Green beads exchange has been for long understood as a key feature of long distance exchange. Recent studies have point to some key elements or element ratios as the clue to define exchange flows. In this paper, we review all the approaches to variscite provenance cross validating them with a dataset of c. 500 XRD, XRF and NMR analysis of the 4 Iberian known outcrops and green bead's assemblages coming from 10 sites coeval to the mines exploitation.

**Keywords:** variscite, provenance, XRD, XRF, NMR

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# A preliminary approach to fluorite beads from the Iberian Peninsula

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Stone beads raw material and shape choices are shown to be much more diverse than expected. Despite the available diversity of colors and minerals in Iberia greenstone overwhelmingly dominates the archaeological record, or at least the archaeological literature. Besides this, research on body ornaments has been unbalanced and highly biased since the beginning, as only rocks which origin can be tracked (variscite and amber) have attracted scholar's attention. Because of this, beads raw material and color diversity has been systematically disregarded, despite many studies have shown an ample variety of minerals used for beadmaking in Prehistoric Europe, e.g. steatite, serpentine, limestone and marble, mica, slate, jadeite, malachite, phosphates, quartz, shell, teeth, ivory, amber, among many others.

Within this plethora of rocks used for the bead making only the pioneering work of Cardoso mention fluorite, and only 5 beads were known for the Iberian Peninsula.

This article presents the first fluorite beads' inventory for the Iberian Peninsula together with a first trial of provenance analysis throughout a p-XRF and p-Raman spectroscopy. We will also focus, fluorite beads spatial variability, chronological and contextual patterning.

**Keywords:** fluorite, beads, provenance, Raman, XRF

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# Understanding the Visual Analysis of Minoan Ceramic Fabrics

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Over a century ago, when Minoan archaeology was a nascent field of research, ceramic vessels were documented in the literature by describing their shape and surface treatment and including photos and drawings when applicable. Gradually over time, more attention was paid to the visual examination of the pottery fabric and painted surface, and more in-depth descriptions of cataloged pottery included Munsell color identifications and details of decoration. Not all scholars, however, adhered to the same terminology. Eventually, the sub-discipline of thin-section petrography was adapted from the field of geology and applied to ceramic studies, whereby microscopy is used to identify stone and other inclusions and their order within the ceramic fabric. Thin-section petrography gained prominence in the scholarship of the Aegean Bronze Age starting in the 1970s, and it has become a routine aspect of integrated archaeological research in recent years. Some hoped that the scientific rigor of petrographic descriptions would help to standardize what had been many and varied nomenclatures for the color and quality of pottery fabrics across Minoan Crete. Petrography has also become an essential tool in providing evidence for the manufacturing process and provenance of ancient clay fabrics. Now is the time to critically re-assess the effect of petrography on the scholarship. We believe it should be used in tandem with macroscopic visual examination of ancient pottery in order to more fully understand the Bronze Age craftsmanship of and trade in pottery. Here case studies will evaluate the published results of petrographic analyses on different types of Minoan pottery.

**Keywords:** Ceramics, thin section petrography, Minoan, Crete, Bronze Age, pottery fabric

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# Regional variation in the use of earliest pottery in Northwest Europe: Case study on Swifterbant pottery and its residue analysis

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This research examines the different early ceramic traditions and the possible functional variation of pottery in the Swifterbant culture in intersite, regional, and interregional scale. Swifterbant culture is a Late Mesolithic-Early Neolithic (5000-2500 BC) hunter-gatherer-fisher society, located in the Netherlands and northern Germany (Lower Saxony), in the NW Europe. Traditional explanations for the introduction of pottery into essentially hunter-gatherer communities consider a direct relationship between (ecologically related) sudden changes in food sources and social adaptation to this change through developing new food processing strategies. Recent studies on the ceramics from hunter-gatherer cultures, however, strongly suggest variance from this theory. Swifterbant culture demonstrates a continuation of hunter-gatherer subsistence strategies alongside a gradual shift towards agricultural cultivation and domestic food production, indicating a shift from the less sedentary life style of hunter gatherers towards the more sedentary life style of village communities that rely on cultivating the land and agricultural production. To illustrate this gradual shift of lifestyle on the pottery and its possible functional variation, this research will work on a selection of pottery from seven different archaeological sites and establish a broader comparative context.

The methods utilized within this research will vary, the primary method will be residue analysis. This will examine the components that have been absorbed into the pottery and through this conclusions about diet, function and variation can be made. Other methods will include morphological and technological analyses of the pottery which will enable a multifaceted understanding of this pottery culture.

**Keywords:** pottery studies, residue analysis, hunter gatherers, NW Europe archaeology

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# The flow of materials in the European Bronze Age: scales, data-classes, and the prospects for integration from a metallurgist's perspective

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The mutability of copper-alloys creates the potential for complex material life stories. It is possible for units of metal to merge, split, be recycled, enter the ground, be reclaimed, and be cast into a series of forms before final deposition. This paper discusses the use of archaeological science to directly characterise processes that have often been the domain of theorists and modellers. Progress has been made on a series of fronts for copper-alloys: new data visualisations, chemical classification systems, quantifying chemical changes caused by oxidation and mixing, and determining the degree and modes of recycling. Overall, it may be possible to build upon a provenance model to understand the socially embedded process of metal 'flow' through prehistory. Even if this work on (some) metals is successful, we are again in danger of describing an unrealistic system: replacing a provenance model with an isolated concept of the way metal moved through society. Instead we ideally want to study how a series of materials, technologies, landscapes, and societies intertwined and interacted. The second half of this paper therefore considers the practicalities of merging the large scientific datasets that are becoming available for metal chemistry and isotopes, stone and ceramic petrography, and organic materials. Key questions that will be addressed include: on what scale is integration feasible, how can GIS-databases be best used, and what primary science is still required?

**Keywords:** Copper, alloy, Bronze Age, Chemical analysis, Recycling, Flow, Database, GIS

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\*Speaker



# Mesoamerican Green Stones Artifacts Characterized by In Situ Spectroscopic Methods: A non-invasive approach.

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Greenstone artifacts were highly appreciated in Mesoamerica due to their symbolic and religious aspects, as well as their physical properties such as color and hardness. Jadeite was particularly chosen for elite groups and ritual contexts. In our group several archaeological burials has been studied from Olmec, Maya, Teotihuacan, and Aztec cultures. The aim of this research is to determine the use of greenstone minerals from Pre-Classic to Post-Classic periods and the raw materials' sources as a first step in understanding the exchange routes in Mesoamerica. In this work, we present a non-destructive in situ study of lithic objects carried out by means of a several spectroscopic techniques, such as Raman, Fourier Transform Infrared (FTIR), X-ray fluorescence (XRF) and color measurements. FTIR and Raman are suitable for the identification of minerals including jadeite, omphacite, amazonite, albite, muscovite and green quartz, among others. Besides, XRF is appropriate for analysis of the elemental composition of the artifacts. This approach allowed to access unique collections in museums and archaeological contexts, and to obtain essential information for sourcing the raw materials, in particular, jadeite. Some outstanding studies of pre-Hispanic pieces are discussed to show the odds and limitations of this methodology. This work has been carried out at the National Laboratory of Sciences for Research and Conservation of Cultural Heritage at Physics Institute of UNAM (LANCIC-IF) by support of grants of CONACYT LN 270749, CB 239609 and PAPIIT UNAM IN112018.

**Keywords:** Greenstones, jade, non invasive, spectroscopy, in situ, minerals, provenance, sourcing, raw materials

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# Analyses chimiques et pétrographiques des céramiques du premier âge du Fer dans le Sud-Ouest de la péninsule Ibérique: bilan et perspectives

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Cette communication présente un bref aperçu des résultats d'un programme de recherche archéométrique qui au cours des dernières années a étudié la composition pétrographique et chimique des poteries du premier âge du Fer du sud-ouest de l'Ibérie, principalement de la région du bas Guadalquivir en Andalousie occidentale. Le but de cette recherche était d'établir une base scientifique pour distinguer entre la poterie phénicienne importée et ses imitations indigènes, d'identifier le transfert de connaissance technique entre les deux parties, et finalement d'établir la provenance des céramiques en question et d'identifier leurs centres de production. A ces fins, un programme analytique a été mené à travers plusieurs collections de musée dans la zone d'étude, en utilisant la technique non-invasive de spectrométrie de fluorescence des rayons X portable (SFXp). Une quantité plus limitée de céramique a été analysée aussi par spectrométrie d'émission atomique (SEA), pour servir d'échantillon de contrôle. Notre méthode principale pour catégoriser les résultats de ce corpus d'analyses a été l'analyse en composantes principales et le test de potassium-titane établi par Y. Goren, H. Mommsen et J. Klinger dans la détermination de la provenance des tablettes cunéiformes.

Les analyses pétrographiques ont été effectuées sur un sous-ensemble de tessons sélectionnés provenant du site de Setefilla (Séville) et comprennent l'examen complexe de la matrice argileuse et des inclusions non plastiques. Les analyses ont été réalisées en adoptant une approche low-tech mais holistique, dérivée de la soi-disant " approche de Leyde ", développée au Laboratoire d'Études Céramiques de Leyde depuis les années 1960, visant à étudier divers aspects de la production, de la technologie, de l'utilisation, du commerce et de la distribution de la céramique. Le principal objectif de notre programme de recherche, consistant à établir des critères qui permettent faire la classification chimique et pétrographique des céramiques orientalisantes d'Andalousie occidentale a été entièrement atteint. D'autre part, seuls quelques sites ont fourni

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\*Speaker

des indications d'une production locale, et nous n'avons pas pu identifier de façon positive des centres de production spécifiques. Nous espérons aborder ces questions de nouveau dans le cadre d'un projet de suivi sur lequel nous nous sommes récemment engagés et dont les premiers résultats seront également présentés dans cette communication.

**Keywords:** âge du Fer, archéométrie, céramique, spectrométrie de fluorescence des rayons X, pétrographie

# Andean connections: a multi-analytical approach to the circulation of materials and artefacts in pre-Columbian NW Argentina

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To explain interaction South-central Andean scholarship has extensively discussed a variety of circulation and exchange practices, with particular emphasis on llama caravan long-distance trade.

While material culture styles and traits were undoubtedly shared, the unexamined focus on similarities leaves the mechanisms, direction, and intensity of interaction to speculation. Provenance analyses can shed new light on these ancient relationships, yet focusing on single analytical techniques obscures the nuances of early interaction.

To further contribute to the detection of the intricate relationships supporting ancient networks, we implemented a multi-analytical approach to different classes of artefacts traditionally considered separately, such as pottery and stone tools. We focus here on the results of the analysis of 542 pottery sherds from first millennium AD sites from NW Argentina, as well as clay samples, obtained through petrography, NAA, and targeted LA-ICP-MS. We discuss these results against the backdrop of the circulation of other materials such as obsidian and other stones, and the complex and long-standing routes they travelled.

In NW Argentina, traditional approaches proposed that regional interaction was an increasingly centralized process, based on typological similarities observed in a variety of materials across the region. Our results provide a platform to examine close intercommunity links rooted on common craft practices, including but not limited to stylistic aspects, and to explore the ancient circulation of goods, skills, and people without assuming the capacity of early elites to manipulate and capitalize on such networks.

**Keywords:** ceramics, obsidian, southern andes, interaction

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# Multi-analytical studies on the manufacturing process of Neolithic ceramics from Jiroft (late 7th - early 6th Millennium BC), Iran

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Jiroft civilization is one of the most interesting places based on the archaeological records. The excavations carried out in this region from 1950, onwards. Metals, arsenical-copper alloys, soapstone, marmoreal objects and ceramics are among the most considerable relics highlighted from this region. 10 pieces of ceramics from Neolithic period (7-6Millennium BC) have been analysed by multi-analytical approach in order to determine the technological features of ceramic manufacturing process in this region.

The samples have been investigated by X- Ray fluorescence for bulk chemical analysis, quantitative X-ray diffraction inclusive Rietveld phase refining method in order to determine the quantitatively crystalline phase constituents. Simultaneous thermos-analysis was carried out for characterizing the phase decomposition during the sintering process and firing temperature. Observation by polarized light microscopy included significant information on the manufacturing process by means of the identification of different additives, used within the ceramic matrix.

The results provided information on the existence of an accidental raw material usage for providing different manufacturing technique in the ceramic production during the 7th and 6th Millennium BC in Jiroft.

**Keywords:** Ceramic, 6th Millennium BC, Manufacturing process, Mineralogy, Crystallography, Rietveld Method

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# Silver in Early Minoan IB Crete

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## Silver in Early Minoan IB Crete

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A program of scientific analyses with X-ray fluorescence (XRF) has been undertaken on the objects from three cemeteries excavated in eastern Crete: Hagia Photia Siteias, Kephala Petras, and Livari Skiadi. All three contexts are dated or have finds that can be dated to the Early Minoan IB period. Among the examined daggers, small tools-such as awls, rods, saws, and chisels-there are also beads, pendants and daggers made of silver. The parallels and the technology of these objects indicate the wide connections of the sites and the developed metallurgical knowledge of EM IB metalworkers. This paper discusses the analyses results in the light of parallels belonging to far away metallurgical traditions.

**Keywords:** Early Minoan IB, Crete, Silver, analysis, XRF, pendants, beads, trade, technology transfer

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\*Speaker

# Pre-Hispanic casting: an approach through Muisca defects fabrication using computational analysis

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Material culture studies in Colombia, specifically metallurgy analyses, represent a challenge to archaeology and related areas of research. The main reason for this is the abundant goldsmith production that survived Spanish conquest, colonization, and late 19th to 20th centuries looting; in other words, archaeologically decontextualized artefacts. Some goldsmith traditions such as Muisca, were developed late, to the point of Spanish contact. Thus, there are some chronicles documenting the meaning of Muisca metallurgy: the vast majority of artefacts had an individual or a small-scale votive offering purpose [1], whereas special artefacts corresponded to major votive ceremonies [2]. Thus, by analysing the manufacturing process of such artefacts not only some production aspects may be evidenced, but also, design decisions that open the debate on the use and availability of metals; that have been long taken for granted. For this reason, we developed an analytical tool along with computational simulation of the casting process of three votive artefacts, through a materials science and engineering methodology.

The computational simulation was based on the reproduction of casting defects existing on the original artefacts, as diagnostic features to validate and understand the pouring process. For this, we obtained the geometries of the objects of study through 3D scanning and used FLOW3D Cast software to simulate the pouring process. We calculated the parameters required on pre-processing based on analytical models, experimental replica procedures, and posterior characterization through XRF, XRD and Optical Microscopy of the mentioned replicas. The pouring simulation and visualization allowed to understand the main parameters creating defects: geometry, mould temperature, amount of metal used, and cooling rate. When analysed altogether, the most likely cause of defect determined was the amount of material, which is directly related to the geometry, which in turn results in a high conductive cooling rate. This suggests that the geometry was modelled taking into account the amount of material available, even if that represented additional challenges on the pouring process; mitigated by mould heating. This phenomena elucidated on three artefacts brings up the question on material limitation, as opposed to the Spanish version of unlimited gold. Also, the evidences act as a call to further alternative analyses that could give more information of the dynamics of the past, when archaeological context is not available.

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**Keywords:** Archaeometry, Muisca, Pouring, Lost, wax, Casting



# The role of SiO<sub>2</sub> and silica rich amorphous materials in the composition of archaeological finds

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The SiO<sub>2</sub> varieties other than quartz of low temperature play an important role in the composition of many archaeological tools, products, byproducts and raw materials. Ranging from Paleolithic stone tools to glass production, moreover ores, smelting slags and finds made of burnt clay belong to the ancient metalworking technologies, we have encountered several SiO<sub>2</sub> species, amorphous or nanocrystalline varieties and also SiO<sub>2</sub> rich glassy materials, which all hide a clue on the type of raw materials, the processing and technological methods or even the usage of the tools. We intend to present several case studies on several kinds of finds (e.g. Paleolithic stone tools, Bronze Age glass products, Iron Age slag or even early medieval iron ore and clay tuyere) to show the optimal synergetic application of mineralogy (X-ray powder diffraction), geochemistry (X-ray fluorescence spectrometry), chemistry (electron beam microanalysis) and microscopy (petrography, metallography with SEM) in unfolding choices and options for the people of many centuries ago.

**Keywords:** SiO<sub>2</sub>, stone, glass, slag, ore, mineralogy, geochemistry, chemistry, microscopy

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# Hypothesis of technological linking between metallurgy and ceramics activities of Tumaco-La Tolita tradition

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The archaeological ceramics of America have been appreciated for its diversity, iconography, style, and uniqueness. However, they have received little attention in interpretative aspects as the organization of production and manufacturing technology. This fact has spread to not recognize the relationship between different kinds of technologies; such as ceramic and metallurgy technologies. Consequently, interpretations of the technology developed in America in pre-Hispanic times are shown as separate activities and developments of a particular social group.

An example of this, are the ceramic and metallurgical technologies Tumaco-La Tolita traditions. The metal objects and potters expressed their artistic preferences and complex technological processes. These technologies have been studied separately and the ceramics are the main sources of the archaeology. Chronology of this tradition has been mainly reconstructed on the ceramics studies. In addition, metallurgy studies have also done independently, moreover the contribution has been much lower for archaeology because most of the objects proceeds of looting or "huaquería" and their studies have expressed the lack of context in collections of ancient metallurgy in Colombia.

Accordingly, this project aims to answer the following research question: Does the evidence in Tumaco's ceramic technology developed in pre-Hispanic times shows several relationships of this activity with the metallurgical technology developed? To answer this question several ceramic figures and metal objects belonging to the ICANH and Museo del Oro Colombia were analysed. The comparison of the operational sequences revealed the potters and metalworkers understood the properties of the materials and its influence on the quality and successful achievement of the final product. Since the results showed selection of the different raw materials suitable for their purposes. These involved decisions of the quantities, sizes, and proportions to obtain mixtures with appropriate characteristics for handling and use. The use of conformation techniques such as: modelling, moulding, compaction of the mixture and plastic deformation that implied strategies reflecting a creative intention. Management, control, and evolution of the pyrotechnology: first, firing of ceramic objects in open furnaces of difficult control and its passage to the necessary use of furnaces and fuels for the control of the atmosphere and temperatures required for metal objects. Finally, the objects analysed showed evidence of finishing operations such as polishing, slip and paint in ceramics and polishing in metals in order to demonstrate the importance in the final appearance of these objects.

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**Keywords:** Metallurgy, Ceramic, Technological linking, Tumaco, La Tolita Tradition

# Approches archéométallurgiques des dépôts non funéraires de la fin de l'âge du Bronze en France (1250 à 800 BCE)

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Le mobilier métallique, constituant des dépôts funéraires ou non, est délibérément délaissé pour des motivations diverses. Pour étudier les dépôts non funéraires, une base de données interdisciplinaire de ces derniers, à dominante métallique, a été réalisée dans le cadre du programme ANR DEPOMETAL et un travail sur les compositions élémentaires du mobilier à base de cuivre a été mené à l'échelle nationale pour la période de 1150 à 800 BCE. Cette étude va poser des questions quant à l'importance des facteurs naturels et culturels au niveau du mobilier métallique des deux complexes culturels atlantique et nord-alpin. Un protocole statistique permettant des regroupements chronologiques et géographiques du mobilier a été élaboré et combiné à un SIG afin de caractériser les compositions élémentaires. Pour la période comprise en 1250 et 1150 BCE, les compositions élémentaires diffèrent de manière significative entre l'Est et l'Ouest de la France. Entre 1150 et 950 BCE, on notera la forte influence de la culture RSFo jusqu'au centre-ouest de la France. Cette observation met en évidence des relations déjà observées pour les périodes précédentes mais jamais démontrées pour ces dates. Entre 950 à 800 BCE, la différence majeure avec la période précédente est le regain des réseaux atlantiques dans le centre-ouest de la France. Ainsi, il semble possible de montrer que le facteur culturel, et donc identitaire, oriente fortement les choix au niveau des réseaux d'approvisionnement et influe sur la localisation des gisements exploités. L'étude offre un nouvel éclairage des réseaux d'approvisionnement pour la fin de l'âge du Bronze en France en révélant l'indépendance des réseaux d'échange en fonction des appartenances culturelles. Le facteur culturel et donc identitaire orientait fortement les choix au niveau des réseaux d'approvisionnement et influait sur la localisation des gisements exploités. Cette conclusion est particulièrement novatrice pour la façade atlantique, souvent présentée encore comme dépourvue de cuivre et dépendante d'importations alpines. Il s'avère que les cuivres utilisés en Bretagne et au Nord-Ouest du Massif Central ont des signatures chimiques différentes des cuivres alpins. Il convient, dorénavant, de déterminer s'il s'agit de cuivres locaux, du Massif Armoricaïn ou du Massif Central, ou d'importations autres que celles du massif alpin.

**Keywords:** Dépôts non funéraires, compositions élémentaires, statistiques, archéométallurgie, Age

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du bronze

## **X-2. Rock-Art Paintings – Binders & Recipes.**

# A MULTI-ANALYTICAL STUDY OF ROCK-ART PAINTINGS FROM MEGALITHIC BARROWS OF THE NORTH-WESTERN PORTUGAL

César Oliveira \* <sup>1,2</sup>, Ana Bettencourt <sup>3,4</sup>, Luís Gonçalves <sup>5</sup>, Maria Isabel Alves <sup>5,6</sup>, André Ribeiro <sup>7</sup>, Alessia Barbosa <sup>8</sup>, Jorge Guedes <sup>8</sup>, Cristina Delerue-Matos <sup>1</sup>

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Some Western Europe megaliths are well known for their paintings. Despite their diffusion throughout Europe, they are most common in Northwest Iberia, predominantly in the Galicia and centre-north and north of Portugal. The rock-art paintings motifs have been interpreted as part as the funeral practices. However, archaeologists are often unaware on both the colorants composition and the technical solutions used on their production, which, also have a symbolic importance.

In that context, the colorant composition of three northern Portugal megalithic barrows decorated with rock-art motifs (Leandro 5, Maia; Leira das Mamas, Braga and Eireira, Viana do Castelo) was studied using a multi-analytical approach. The use of XRD, SEM-EDS and FTIR combined with GC-MS allowed the characterization of the painting techniques, pigments, and the organic compounds used as binders.

The analytical results obtained allowed the recovery of important data about North-western prehistoric communities, namely the way they exploited existing resources and their ability to transform them.

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\*Speaker

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**Keywords:** Megalithic barrows, Pigments, Binders, Chemical analysis, Gas Chromatography coupled with Mass Spectrometry (GC/MS)



# Experimentation of rock art pigments in order to increase the m-FTIR spectrum databases of organic and inorganic paintings material

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An experimentation project in order to better understand the different shades of red and its chronological subdivisions of rock paintings from the Bovidian period, Central Sahara was made. Within a prehistoric context the colour red/yellow/black/white have been the popular choice among artists. The use of red (and various shades of) is found on rock panels across most the world. Within several areas, artist considered the concept of producing polychrome images whereby allowing red and its various shades to create movement, proportion and perspective in their paintings. Currently knowledge shows that the artists were engaged in a process of mixing both organic and inorganic substances together in order to achieve the desired pigment colour. However, organic material is not easy to identify and m-FTIR (Micro-Fourier Transform Infrared Spectroscopy) is a technique that is being used due to the organic recognition possibilities that presents. The recipes used in the experimentation will update a database that will provide a better understand of the different shades of red and its stylistic subdivisions of rock art paintings from the Bovidian period in Central Sahara -  $7327 \pm 25$  BP (Ginette, 2006), as well as the identification and use of binders in recipes elsewhere. The sampled substances analyzed by m-FTIR contain different iron oxides and hydroxides (heated or not), charcoal, graphite, kaolin, olive oil, animal fat, cactus sap, egg yolk, human blood, and other materials. Through Archaeometry study, applying an experimentation approach, it will be possible to evaluate the organic binder substances used in the production of prehistoric pigments. The samples of inorganic and organic paintings recipes were used to create several m-FTIR spectrum databases that will assist the known data of the various constituents that are used in prehistoric rock-art paintings.

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\*Speaker

**Keywords:** Rock, art paintings, prehistory, experimentation, pigments, binders.

# Identification of organic binders in prehistoric pigments through multiproxy archaeometric analyses from the Toca do Paraguaio and the Boqueirão da Pedra Furada shelters (Cativara Sierra, Piauí, Brazil).

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Pigments from several prehistoric rock art paintings samples were analysed through a multiproxy archaeometric approach. Sampling occurred in two rock-shelters within the National Park of Serra da Capivara area (an UNESCO World Heritage Site): The Toca do Paraguaio and the Toca do Boqueirão da Pedra Furada.

Five complementary techniques such as X-Ray Micro-Fluorescence, Raman Spectroscopy, Scanning Electron Microscopy, Stereomicroscope Observation and High Temperature-Gas Chromatography were combined in order to identify and characterize the inorganic and organic pigment materials. In particular, gas-chromatography analyses revealed the presence of binders, hardly recognized in rock art prehistoric pigments. These binders' substances (plant fatty acids) could be included in the pigment process "chain-opérateur".

**Keywords:** Organic, binders, Brazil, paintings

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\*Speaker

# Identifying temporal and regional trends in ochre selection, acquisition, and use at the Upper Palaeolithic cave site of Hohle Fels, Germany

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Here, we present the results of an investigation of ochre artefacts from Hohle Fels cave in southwestern Germany. Ochre use is well documented at the site with numerous painted limestone fragments and modified ochre pieces. New insights from a re-analysis of the assemblage revealed 24 other anthropogenically modified ochre artefacts, as well as 864 non-modified ochre artefacts from the entire Upper Palaeolithic sequence (12.5-40ka BP), as well as the Middle Palaeolithic layers. The ochre pieces were classified by various qualitative criteria, including color, size, and streak, and visual assessment indicates clear changes through time in patterns of source exploitation and utilization. Ochres collected during the Aurignacian (32-40ka cal. BP) are rarely modified, and consist of lighter red varieties with larger grain inclusions. Preferences change in the later Aurignacian layers and Gravettian transition layers. The Gravettian (27-32ka BP) and Magdalenian (12.5-13.5ka BP) assemblages, which contain more anthropogenically modified pieces, show a shift in preference towards more hematite-rich dark purple pieces. Many examples contain micaceous inclusions, are finer-grained in texture, and produce dark-red streaks. The modifications on the artefacts from these time periods are consistent with pigment extraction, i.e. macro- and micro-striations. There are also numerous other artefacts from the Gravettian and Magdalenian containing traces of red residues, including faunal elements, shells, limestone fragments, and personal ornaments like tooth and ivory beads. The diachronic trends in ochre selection and acquisition were further investigated with the use of neutron activation analysis (NAA) in order to identify trends in geological selection throughout the Upper Palaeolithic. It is our hypothesis that ochre collection during the Aurignacian was more localized and

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expedient, and shifted over time to include more rare and exotic sources from other areas of southern Germany and possibly farther. Furthermore, we conducted a comparative geochemical analysis of samples from nearby contemporary cave sites of Geißenklösterle and Vogelherd to assess the potential for networks of ochre trade or shared use of ochre resources in the region. This study promises to shed light on further aspects of the acquisition of artefacts used in symbolic capacities in southwestern Germany during the Upper Palaeolithic.

**Keywords:** Red ochre, Upper Palaeolithic, Pigments, Symbolic behaviours, Human, mineral interactions

# Limites et possibilités de l'identification et de la caractérisation des agglutinants des pigments levantins : la région de Valltorta-Gassulla (Castellón, Espagne) comme cas d'étude.

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L'art Levantin est une manifestation pariétale unique à l'échelle de la préhistoire européenne. Malgré le potentiel d'information que renferment ses scènes, par la représentation d'activités économiques et sociales et de la culture matérielle, à ce jour le contexte chronologique et culturel de ces peintures reste inconnu, car il n'existe aucune datation des composants organiques des pigments par le radiocarbone. Si l'identification récente de charbon de bois comme matière première principale des pigments noirs des abris ornés de Gassulla (López Montalvo et al, 2014 et 2017) ouvre la possibilité d'obtenir les premières datations directes des peintures, la quantité de matière nécessaire et les problèmes de contamination associés aux systèmes en plein air rendent l'obtention des datations par le radiocarbone inenvisageable à ce jour. Il en va de même pour l'identification et la caractérisation des substances organiques utilisées comme agglutinants lors de la préparation de ces pigments. Susceptibles également d'être datées, leur faible quantité et/ou leur dégradation suite à l'activité des microorganismes ne favorisent pas leur analyse par des techniques traditionnelles.

Dans cette communication nous présentons les résultats des analyses protéomiques développées dans les abris levantins de Valltorta-Gassulla (Castellón), en évaluant en même temps les limites et les possibilités que nous offrent des nouvelles techniques d'analyse testées dans cette région.

**Keywords:** Art Levantin, Néolithique, Valltorta, Gassulla, pigments, agglutinants, datations radiocarbone, analyse protéomique

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# On Palaeolithic coloring agents: epistemological analysis of scientific process

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What were the coloring agents used during the Palaeolithic? How to recognize, define and describe them? What kind of information may they have recorded? In the past decade, these questions have become essential since coloring agents take a central place in current researches on modern human behaviors and emerging of figurative expressions. Typically, coloring agents are found in various archaeological/sedimentological/economical/cultural contexts: open air and rock-shelter settlements, graves, drawings and paintings in caves.

As a consequence, this topic needs interdisciplinary approaches. There is a crucial challenge to overcome which starts from sharing semantics adapted from geosciences, archeology and material sciences. This allows to strengthen the fundamental knowledge to build the working hypothesis and the methodology.

Notably, as for the flaked industry, the "cha ne op eratoire" (processing sequence) analysis has proven to be an efficient tool. It provides i) a path to a dynamic comprehension of coloring material processing and ii) a way to include this productions within a systemic framework of Palaeolithic activities. Applied to the coloring agents, it concerns:

1- The qualification of their sensitive (visual and mechanical) properties: color tone, texture, proneness to be ground to powder.

2- The procurement strategies which require the setting up of geological references (Project *Pigmentothenque*), and combine information concerning geological contexts, availability and sensitive properties. With a combined and reproducible documentation and characterization procedure of the raw coloring rocks, we **identify the geochemical fingerprint** with different scales of information (petrography, mineralogy, geochemistry). This gives robust data in order (i) to compare raw materials to coloring agents used during the Palaeolithic, even if they were transformed by heating or mixing, as well as if they were ground (ii) to powder, or strongly modified through taphonomic processes.

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3- Techniques implemented to use the coloring agents are corollaries of sensitive properties, supply strategies, as well as the objectives of the production. Thus, techniques are systematically documented (powder production, hue control, grain size of the powder, mixing with other substances).

4- As a synthesis of the three previous stages of analysis, the objectives of the production are questioned with regard to sensitive, availability and cultural concerns.

At this point, by studying current scientific methods and discourses on past human activities related to coloring agent exploitation, we can focus on the possible weaknesses in the scientific procedures.

**Keywords:** Epistemology, Colouring agents, Palaeolithic, Interdisciplinarity



# Rock art in the Chicamocha Canyon, Colombia

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This study describes rock art panels located in the Northeastern area of Colombia especially in the Chicamocha Canyon, which is considered the second largest seismic nest in the World. Spanish chroniclers described that area was inhabited by indigenous communities called as *Guanes*. Archaeological research aims to know how this prehispanic society thrived in this particular territory. For instance, the geological formation features a wide range of pictographs which vary in design: human like shapes, animals and plants designs as well as a wide range of colours: ocher, orange, white, black, cream in monochromatic and bichromatic drawings.

Most efforts have been orientated in the description of the rock supports, motifs and weathering, in order to identify criteria of rock art distribution and preservation. So far, fifty-seven panels have been recorded *in situ* following a systematic survey that aims to elaborate a baseline from which further studies can be developed to answer some of the following inquiries. First, relationships between the type of motifs and its location over the rock. Second, distribution of rock carving within the landscape particularly water resources. Third, list of main factors of weathering for finding measures of preservation. Fourth, identification of painting techniques and pigments used. Fifth, classification of rock supports where drawings are located, to mention some of them.

Finally, the study wants to contribute with the understanding of rock art in Colombia and the American region by engaging archaeologists and other kind of in the protection and promotion of this cultural heritage.

**Keywords:** Rock art, Chicamocha Canyon, Guanes, Colombia

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# Rock painting elements reassembled by using spatial metrics

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Rock paintings exposed to bad weather conditions, especially when they are on bare rock faces, deteriorate progressively and it is often necessary to gather the elements that belong to a same figure to study it. It is the case of the Mezquital cave paintings in Hidalgo state (Mexico). The software COATL recently developed by two of the authors can be used to reconstitute the rock drawings, to estimate the degree of degradation, and then, differentiate the different families of paintings. The software selects automatically the pixel components that belong to a given shape, labels them and extracts each component perimeters to unite these elements according to the distance that separates them. The reconstituted figures can be then submitted to a series of test and measurements such as surface and/or perimeter length calculations, convex surface inclusions, fractal dimension, etc., without the purpose to realize a classification based on characteristic features. A set of different style paintings has been studied to estimate the confidence rate of the proposed treatments; these treatments concern mainly the enhancement of the drawn features by means of a new kind of thresholding, a gray tone exploration providing a controlled extraction of the item to be studied, a sorting of eventual superposed structures that can be then independently studied, the calculation of indices based on objective relationships between shape perimeters as well as between shape surfaces, the integration of the results in a data base allowing to compare the characteristics of the painting families. Our aim is to develop and propose a general technical approach that can be applied in different archeological contexts.

**Keywords:** Rock paintings, digital image processing, spatial metrics, shape indices

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# The first results of experiments with haematite for reconstruction of the Upper Palaeolithic art practices in Kapova cave

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Kapova ave is located in the Southern Urals (Russia). Upper Paleolithic wall paintings represented by figures of mammoths, horses, rhinoceros, bison, fish, camel, zooantropomorph and geometrical signs. They are mostly made with ochre of different shades of red.

A series of experiments were conducted for obtaining a pigment's colours, similar to that were used in Kapova ave to create the wall paintings for the reconstruction of Upper Paleolithic art practices. The raw materials were brown iron ore (haematite), the deposition of which are located nearly 2 km from the cave. This same raw material was used by the artists of the Upper Paleolithic in the Kapova cave, which was revealed by the results of the analysis of the pigment of the drawings and the pigment from the cultural layers.

Selected samples were burned in order to get equivalent examples of ochre. Such processing methods as crushing and adzing the same piece of haematite were used. Pigments were obtained that differ in color and intensity. One of the most important results, from the point of view of comparison with materials from the Upper Paleolithic cultural layers, was the identification of raw material processing technique, the traces of which can be seen on the materials from Kapova cave and Talitsky's site. The observed result was obtained by grinding by stone a piece of haematite placed on the tile (after a preliminary crushing of the lateral part of this stone into smaller pieces). Traces of the paint were very few and barely noticeable on the lateral side, but on the side which we triturated haematit, there was a very large amount of ochre, embedded in the surface of the stone and not being scraped off by shaking. It is possible to assume that such technique (or similar one) were used to make the coloring pigments, which then were used in artistic practices.

It has also been experimentally confirmed that the color of the pigment varies depending on the method of processing the original raw materials, and not only on additional ingredients which mix directly with the main pigment.

The results of the study of possible techniques for processing raw materials for obtaining different colors and shades of coloring matter, as well as processing traces that can be observed on the (lime)stones after the experiment, permit to identify better the separate functional zones of the underground cave with wall images of the Upper Paleolithic.

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**Keywords:** Stone Age, Upper Palaeolithic, art practices, Kapova cave, experiment, haematite, ochre, wall paintings, reconstruction, pigments