PHYSICAL ANALYSIS OF SEDIMENTS
Paleosol in archaeological context
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Abstract

My research project was centered on using physical methods to analyse paleosoil for an archaeological purpose. I have chosen to present in this report the two main projects on which I have worked during the month. Though they both dealt with prehistoric times in Europe, they spanned over different periods of the archaeological record: if the first was about Early Bronze Age in Cornwall (2500-1500 BC), the second covered a wider older epoch, the Last Termination (the transition ca 20000 to 10000 BC between Late Glacial, the last glacial stage in the history of Earth, and Holocene, the current interglacial stage). The physical analysis was of several kinds: X-ray spectrometry for elemental analysis (X-ray fluorescence and microprobe analysis), electronic microscope (SEM), mineralogy and geophysics.

The aim of the first project was to look for evidence for an early development of metallurgy in England: finding tin in the soil samples on which I worked could prove that in the Early Bronze Age Cornish people knew already how to smelt tin metal separately of copper.

The subject of the second project is related to one of the major current issues for Quaternary research: establishing a precise chronology of events for the Late Glacial-Holocene Transition more than 10000 years ago in order to understand climate change and its correlation with human evolution. For that purpose, radiocarbon age sequences are worked out with help of teprochronology (volcanic layers and the composition of their tephra are used as precise chronological horizons) and Bayesian analysis (data modelling).

Résumé

Mon stage portait sur l’utilisation de méthodes physiques pour analyser le paléosol dans un but archéologique. J’ai choisi de présenter dans ce rapport les deux projets auxquels j’ai participé durant le mois. Bien qu’ils concernent tous les deux l’époque préhistorique, le premier se rapporte à l’Âge du Bronze en Cornouailles (2500-1500 avant JC) tandis que le second couvre une période plus vaste et plus ancienne (la transition de 20000 à 10000 avant JC environ entre la dernière ère glaciaire et l’Holocène, ère interglaciaire actuelle). Les analyses physiques réalisées sont de différentes natures: spectrométrie rayons X pour l’analyse élémentaire (fluorescence X et analyses à la microsonde: EPMA), microscope électronique (SEM), minéralogie et géophysique.

Le but du premier projet était de chercher des indices d’un développement précoce de la métallurgie en Angleterre: la découverte d’étain dans les échantillons de sol sur lesquels j’ai travaillé pouvait mettre en évidence que la technique de la fonte de l’étain (séparément du cuivre) était acquise en Cornouailles à l’Âge du Bronze Ancien.

Le sujet du second projet s’inscrit dans l’un des principaux enjeux de la recherche actuelle sur le Quaternaire. Il s’agit d’établir une chronologie précise des événements de la transition entre le dernier Âge Glaciaire et l’Holocène il y a plus de 10000 ans pour comprendre les changements climatiques et leurs conséquences sur l’évolution de l’Homme. Pour cela, on se sert de la teprochronologie (qui consiste à prendre comme marqueurs chronologiques précis les couches de cendres volcaniques caractérisées par leur composition géochimique) et de l’analyse bayésienne.
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1 Introduction

The purpose of the research project that I carried out in the Research Laboratory for Archaeology and the History of Art (RLAHA) in Oxford was to apply physics to archaeological issues. In effect, many questions in the research in archaeology can not be answered without physical and chemical analyses, and that is even more true when the period of interest is very ancient and so when historical data are missing. Due to a lack of written records in prehistoric times, physical and chemical sciences are particularly essential to a better understanding of human evolution and the question of the development and adaptation of Humans to a new environmental context (often because of climate changes).

Human science, by studying the stylistic features of archaeological artefacts, by taking their historical context into account, and thanks to the comparison with old textes, provides answers for many questions about human history. However precious informations about their origin, the way they were produced and their age can be infered from scientific analyses (giving the physical and chemical properties of the materials) such as the determination of the mineralogic, structural and elemental composition of the archaeomaterials. The study of archaeomaterials is a science in itself because the structure of the matter is altered through time due to several factors such as human intervention and weathering. Analyses must be performed at different physical scales. More and more, thanks to the improvement of physical and chemical techniques, analysis of microsamples in archaeological context has been developing. To mention only a few examples, from less than three grammes of human hair, the paleodiet of our ancestors can be infered; the geochemical composition of microtephra (volcanic ash) can be identified by using a microprobe; tiny beads of soil sediments can be observed with an electronic microscope and dated one by one...

For my research project I performed various analyses on soils in archaeological records. But in my report I have chosen to concentrate on two of them. The first, whose context is Early bronze Age in England, is the search of tin in three samples from Cornwall. I worked with a research student, Peter Bray. He is working on a PhD that focuses on tin-bronze in Bronze Age in England. The second period in which I took an interest is older: it is the Last Termination, the transition at the end of Quaternary from the last Glacial Stage to Holocene. A researcher of the tephrochronology group of the laboratory, Dr Simon Blockley, supervised this project. Since 2004 and the set up of the micro-tephra extraction laboratory, the main research interest of the tephrochronology group has been to establish micro-tephrochronology as an archaeological dating tool particularly for sites in UK, Europe and North Africa. This research contributes (inter others) to the expansion of the known database of European micro-tephra deposits and eruptions of the geological record and to the construction of precise chronologies for archaeological and environmental sites in order to understand for example correlations between climate change and human development.

2 Tin in Cornwall

2.1 Context and stakes

The project dealt with Early Bronze Age in Southwestern England. In Northwestern Europe, this period extends from about 2500 years BC to 1500 BC. At the end of the Neolithic, people began to use metals. First they learned to produce copper by smelting copper ore (that contained often impurities). Then, with the improvement of metallurgy, tin was added to copper and most of the Bronze Age artefacts (axe’heads, daggers, pins) were made of alloys such as bronze (traditionally 10% tin, 90% copper) whose properties had revealed to be better from a point of view of efficiency as much as aesthetic.

The technique that was first employed to make bronze consisted of mixing and smelting the copper metal with cassiterite (a tin oxide : the tin ore that they extracted from mines). This was probably discovered by accident because of the impurities included in the ores they used to
smelt copper. They produced copper metal from the ore \((\text{Cu}_2\text{O} + \text{CO} \rightarrow 2\text{Cu} + \text{CO}_2)\). Then they added cassiterite to smelt the bronze: \(\text{molten Cu} + \text{SnO}_2 + 2\text{CO} \rightarrow \text{Tin} – \text{bronze} + 2\text{CO}_2\). Later, they became able to smelt tin metal separately: \(\text{SnO}_2 + 2\text{CO} \rightarrow \text{Sn} + 2\text{CO}_2\). Thus they could export the two metals and a smith mixed them in the furnace just for making the artefact.

The precise chronology of this improvement of the bronze industry can’t be established by referring to historical texts because there is no trace of writing at that stage in England. Typology studies (classifying artefacts according to the material used in the shape and design) have been carried out, but they generally don’t allow to date exactly the events and the fact that the artefacts that are found during excavations could have been imported from other places of Europe and not produced in-situ makes the task difficult. That is why science is necessary to reconstitute the precise chronology of the development of metallurgy in England.

2.2 The project

I worked on three soil samples from a site in Cornwall in the westsouthern part of England. Each one had already been radiocarbon dated. The context sample was as follows:

- The first came from a hearth of Early Bronze Age.
- The second was found in a pit of Early Bronze Age. Although the deposit was charcoal-rich, there was no evidence of burning in-situ.
- The third sample was sent to the laboratory as a control sample : it was a piece of a medieval buried soil.

As the samples did clearly not contain copper but hopefully tin, the purpose was to analyze them in order to find if there was tin in them and if it was the case, to determine which chemical form tin appeared. The presence of tin in one of the two first samples could prove that people at that time already knew how to produce tin metal for itself. The samples could also contain cassiterite as trace of the initial ore. Though tin metal might be expected only in very small quantities because the smelted metal was probably removed to be used as ingot or artefact, we hoped to find slag, a waste product of the activity of metallurgy. In Caerloggas (a cornish site from the Bronze Age whose context, barrows of the Saint-Austell granite, is really close to the context of our samples), tin smelting slag has been found as a black glassy phase with globules of metallic tin.

However, if a flow of slag usually forms in a furnace (made of clay and stone and where the temperature can be very high), there can be very little gangue (only small droplets of slag) produced when metal is smelted in a bonfire (due to low temperature). And no furnace structure dating back to the origins of metallurgy has been discovered in Atlantic Europe, as a result of which the first smelting places are supposed to be bonfires. Hence we did not expect to find tin-slag in big amounts in our samples more especially as slag is a glass that easily breaks down.

2.3 First observations

The samples were first observed with a microscope. They were sieved to separate the grains by size: three categories were distinguished (> 500 microns, > 63 microns, > 53 microns). If there was tin in the sample, as the expected quantities of metal were not high, it would be easier to find it after sieving, because tin would probably most appear in one of the three grain-sized bags depending on its form (metal, ore...).

A first observation and attempt of separation of the grains according to the colour revealed different types. The two first samples contained black grains easily identified as charcoal : the first came from a hearth and the second was probably a pit where remains of a fire had been buried. A few shining beads in the samples suggested traces of metal. In the samples there were also grey, yellow and red grains -including normal earth in a granitic context and maybe dark pieces of slag.
A separation by density seemed to be a more efficient means of isolating tin (than separation by colour) because of the high density of the metal \((6.99 \text{ g/cm}^3)\). The method is quite simple. A few grammes of the sample are diluted in a solution of SPT (Sodium Poly Tungstate) with a density of \(2.6 \text{ g/cm}^3\). The test tube is put in a centrifuge (that must be cautiously balanced before running) for 15 minutes. Two phases appear in the tube. After eliminating the light (charcoal-rich) fraction, the heavy fraction (that might contain the metal) is washed five times in order to get rid of SPT: the grains are diluted in distilled water and put (after shaking) during five minutes in the centrifuge, then only the grains (that stay at the bottom) are kept (the water is eliminated). To finish the procedure, the cleaned grains of the heavy fraction are dried in an oven and put in a Pettrie dish.

2.4 XRF-analysis

X-ray fluorescence spectroscopy is one of the fastest and most frequently used techniques of elemental analysis. A beam of X-rays (primary X-rays) is emitted by an X-ray tube and irradiates the sample. Due to the high energy of these rays, innermost electrons of the sample are ejected by absorbing the energy of the striking X-rays (photoelectric effect), creating vacancies. As the atom has become unstable because of these vacancies, electrons of the outer shells can then deexcite by emitting secondary X-rays of precise energy (it is the difference between the two binding energies of the corresponding shells). As each element has a unique set of energy levels, the energies of the secondary X-rays are characteristic of the elemental composition of the sample. Detectors of energy count the emitted photons of each energy. The spectrum that appears on the connected computer screen shows peaks corresponding to the secondary X-rays and thus to the elements that are present in the sample. The size of a the characteristic peaks depends on the amount of the element contained in the sample. And for one element, the proportion of the different peak-sizes is always the same.

Although this method is not very precise and more qualitative than quantitative, it has the advantage of being non-destructive and provides a good means to detect easily and quickly the presence of an element (an analysis by XRF takes only a few minutes) in any form of sample.

For our study, we put in the beam a few grains of each of the nine grain-sized samples (one sample by one). The spectra that we obtained were compared to a spectrum of a standard 10% tin-bronze where the expected peaks characteristic of tin appear clearly: \(K_{\alpha1}\) and \(K_{\alpha2}\) at 25keV, \(K_{\beta}\) at 28 keV, \(L_{\alpha}\) at 3keV, \(L_{\beta}\) and \(L_{\gamma}\) at 4keV. (the letters \(K\) and \(L\) refer to the shell of the vacancy while Greek letters indicate the original shell of the deexciting electron). And copper produces two peaks around 8keV.

First we have to notice a few characteristics that are the same for all the spectra of our samples. The lack of peaks at 8keV confirmed the absence of copper. Two big peaks at 6keV reveal the high iron content of the soil. The peaks around 3keV, that can be seen on the spectra even when there is no tin-peak at 25keV, stand probably for calcium. The interpretation of the spectra must be led carefully because the presence of one peak corresponding to one transition between two levels of a particular atom does not always imply the presence of the element in the sample: only the presence of the entire set of expected peaks can be interpreted as characteristic of an element. There is indeed much noise and sometimes supplementary peaks can appear because of parasite reflexions.

There are also two peaks around 21keV. They are characteristic of the rhodium \((K_{\alpha}\) and \(K_{\beta}\)) that is the main constituent of the X-rays source and do not depend on the composition of the sample. One big peak at 19 keV does not seem to correspond at any element that could be expected in a soil sample: the cause might be an effect of Bremsstrahlung. At last, the apparent presence of calcium responsible for a peak around 3keV and the surrounding noise did not allow to detect a peak caused by the \(L\) shell of tin. Hence traces of tin could only be revealed by a peak at 25 keV, which might be a source of uncertainty. But if we consider a quite large interval around
Figure 1: X-ray spectrum of sample 2 showing a peak at 25 keV characteristic of tin.

this value of energy, tin is the only element that can emit in this band. Therefore it can not be
confused with another element.

The results were not exactly what we expected. The first sample did not seem to contain
tin. On the contrary, the control sample was clearly full of tin - the tin peaks were big for the
middle-sized grains, a bit less big for the big-sized grains (and did not exist for th small-sized
grains). In the middle-sized fraction of the second sample, one of the three analysis that we carried
out was positive, with a clear peak at 25keV (see figure 1). We had probably once the chance to
put one of the seldom grains of tin in the X-ray beam, because the two other spectra were exactly
the same as the first apart from this peak at 25 keV.

The third sample, sent as control sample, had apparently not been well chosen, because it
should not have contained tin. The explanation of the rich content in tin of this medieval sample is
probably the fact that soil had become tin-rich in the Middle-Age due to the massive development
of the metal industry.

We noticed that tin was systematically absent in the small-sized grains. Actually, the fine
fraction of the samples (between 53 and 63 microm) was simply dust. Cassiterite beads or slag
droplets, if present, were bigger than 63 microns.

There was now evidence of tin in the middle grain-sized fraction of the second sample. We
decided to carry out further analysis in order to determine which form of tin it contained.
2.5 Mineralogic analysis

We tried to make precise observations with the microscope in transmitted and polarized light. We set a few grains of the middle fraction of the samples on a lame with a non fluorescent fractoil to make observation easier. We observed the samples and compared them to a reference: crushed pure cassiterite. The light was polarized. Thanks to the addition of a second polarizer, whose polarization is perpendicular to the first, minerals can be distinguished. They are the only grains visible when the two polarizers are set: they appear colourfull and the colours, changing with the rotation, depend on the nature of the mineral through its birefringence.

Without the second polarizer, cassiterite usually appears as dark (brown, sometimes yellow) grains showing right angle cleavages and with well-defined contours due to a high refractive index. Cassiterite has to be distinguished of tourmaline - the observation showed tourmaline impurities in our samples. Tourmaline is also a brown mineral, but its contours have less intensity because of a lower refractive index. According to the way of mineral growth, tourmaline presents cores (circular growth) whereas cassiterite shows straight lines (linear growth). By rotating the sample, the colours of cassiterite might change but can not include green contrary to tourmaline that often becomes green. With the second polarizer, cassiterite and tourmaline appear colourfull (they are both minerals). The colours of cassiterite can be very bright (with blue, red...).

However cassiterite was not easily distinguished from the brown minerals of a normal granite. Following the advice of a geologist, we decided to observe the samples on reflective light (with the microscop). Therefore we fixed a few grains of the soil in a resin (by using a vacuum chamber) and we polished the surface of the resin where the samples outcropped. Then we observed them using reflexive light. Metallic minerals should now appear as shining beads. We found in the second sample tiny irregular grains (probably glass) with shining points (maybe tin) and full of bubbles that can be the traces of liquid stuff: hopefully it was tin-slag.

3 Tephrochronology and radiocarbon dating of Quaternary sediments

Another aspect of analysis of sediments is the tracing of tephra in the environmental records.

3.1 Quaternary and climate change

The Quaternary is the more recent period on the geological timescale. It is usually supposed to begin 2.5 million years ago (and to extend until today). Known as "Ice Age", it has been a succession of glacial (cold) and interglacial (warm) stages. The period of interest for my project was more precisely the end of the Quaternary: the transition between the last glacial period, Late-Glacial (15000-11000 cal years BP), and Holocene (the last interglacial period that extends from around 11000 cal years BP to nowadays). As it is the most recent period during which such rapid high-amplitude climatic fluctuations occured, it is essential to establish a precise chronology of events for that epoch in order to have a better understanding of climate change in general and at that period in particular - because it corresponds to the beginning of huge human dispersal and evolution and it could have played a prominent part in this process.

Thus, one of the biggest issues for current research in Quaternary is to determine whether the fluctuations of climate were synchronous during the Late-Glacial transition in Europe and North Atlantic. As significant temperature variations take place within a few decades, it is therefore necessary to be able to rebuild very precise chronologies. The most used dating method is radiocarbon dating. But the exact reconstitution of the chronology of a given sequence of successively deposited sediments is difficult due to lack of both accuracy and precision in the dates obtained compared to the scale at which the climate system operates. There are overlaps and reversals. I have worked
on two means used to overcome these difficulties. One of those is tephrochronology that is very useful because many sequences contain tephra layers. The second is a particular way of working out data and probabilities: Bayesian analysis.

3.2 Tephrochronology

Tephra layers have been deposited during volcanic eruptions. Thanks to the analysis of the physical or chemical properties of tephras, the volcano and the eruption that produced this layer can be identified. Thus the age of the tephras can be determined because of the database that was built up by using different methods of dating and various sequences. As the deposition of the tephra (emitted material: bombs, lapilli and ash) is essentially instantaneous on a geological timescale, these layers provide distinctive and often widespread isochronous marker horizons for establishing chronologies.

3.2.1 Microtephra analysis: electron microprobe

Far from the volcano, traces of an eruption can be found as microtephras (distal ash). The chemical composition of volcanic glasses can be determined by electron probe micro-analysis (EPMA) and used as geochemical fingerprinting to identify the tephra.

The principle of the electron microprobe analysis is very close to the XRF-analysis. The sample -tephra sherds fixed in a resin- is bombarded by an electron beam. Electrons are thus removed from inner shells (secondary electrons) and electrons of the outer shells can deexcite by emitting X-rays whose energies are characteristic of the element. Contrary to XRF (where energy-detectors are used), the X-rays are separated according to their wavelength by dispersion by a crystal. Therefore each element can be precisely and separately detected.

Additionally, the electron microprobe can function as a scanning electron microscope (SEM) and obtain highly magnified images of the sample by detecting secondary electrons. SEM can be also used for targetting on very small individual shards. That is the reason why EPMA is considered a precise spot analytical technique.

However EPMA presents a few problems. If the bombarded surface of the tephras is not very flat, the results may be wrong. That is why tephra are first fixed in a resin and the surface has to be grinded (to make the grains appear at the bottom of the resin) and then polished with diamond pastes (of decreasing grain-size) which, as I experienced it, takes quite a long time. But it is absolute necessary to get good data.

I took part in the targetting process. The points have to be chosen right in the middle of tephra sherds that appear shining (because glass is a material that can be very well polished) and that include bubbles (during the cooling of tephras, bubbles of gas are trapped in the shards). And the position of the crystal must be adjusted (with the help of an optical microscop) to provide accurate results.

So 50 tephra sherds are targetted successively (during a few hours) in order to assess the chemical range of the volcanic glass. Then can begin the task of identification of the tephra layers.

3.2.2 Identifying the tephra by their geochemistry

I worked on a sequence of radiocarbon ages from Ireland where tephra had also been identified. It was divided in three main parts:
- one from a depth of 52cm to 26cm (the older part) corresponding to Late-Glacial deposition.
- one between 26cm under earth and 2cm over with only one radiocarbon date but containing two tephra layers refered to as tephra A (depth: 25cm) and tephra B (depth: 12cm). The geochemistry of the tephra had been determined by using EPMA.
- the last between 2cm and 10cm over earth from the beginning of Holocene. The purpose was to build up a precise chronology for this record. Here intervenes tephrochronology: using tephra layers as markers. I had to identify tephras by their geochemistry.

Ten major elements (expressed as oxides) were given for a series of samples belonging to tephra A and tephra B. I had at my disposal a databasis of the possible sources: the chemical composition of characteristic tephra from the main eruptions of the Last Termination and Holocene. The active volcanoes at that time in Europe were essentially divided in four areas: Iceland, the Eifel district in Germany, the Masif Central and Italy.

To compare the data properly, binary diagrams of different oxides are plotted for the different tephras including those of interest. Tephra A were easily identified with a TAS diagram (total alkalis-silica diagram) as belonging to Laacher See tephras because this eruption of Germany was very important and had a characteristic easy identifiable geochemical fingerprinting. The TAS diagram, that represents the sum $Na_2O + K_2O$ against $SiO_2$, divides rocks according to their silica content into basic, intermediate and acid (see figure 2). Further analysis using plots of different elements ($Mg$, $Al$, $Ti$...) versus $SiO_2$ (Harker variation diagrams) proved that tephra A were part of Middle Laacher See tephras that had been dated by varve chronology: 10930 ± 50 BC.

Tephra B were not as easily identified as tephra A. A preliminary TAS diagram allowed to keep only five types of tephras corresponding to five possible sources for tephra B. A clear identification resulted from plotting of bi-diagrams showing elements as $Fe$ or $Ti$ vs $Mg$: tephra B belonged to Vedde Ash (from an Icelandic eruption). The age of Vedde Ash is known thanks to Greenland ice-cores records (GRIP age): 10030 ± 60 BC.

Figure 2: TAS diagram showing tephra A (red circles) in the middle of Laacher See tephras.
The second part of the work was the construction of the chronology itself by using Bayesian analysis (as explained further) and by including (after calibration of the radiocarbon ages) the dates obtained for the tephra layers.

3.3 Working out data and modelling

3.3.1 Radiocarbon dating and problems of calibration

Radiocarbon dating is based on the measure of changes in the isotopic ratios of carbon due to radioactive decay. $^{14}\text{C}$ decays via the emission of a beta-particle (electron) to form $^{14}\text{N}$ that is a stable isotop of nitrogen. Radioactive decay is exponential and the half-life $t_{1/2}$ (period of time required to reduce a given quantity of parent nuclide by one half) depends on the element. For carbon: $t_{1/2} = 5730 \pm 40$ years.

$^{14}\text{C}$ is permanently formed in the upper atmosphere through the interaction between cosmic ray neutrons and nitrogen. The atoms produced combine with oxygen to form $^{14}\text{CO}_2$ which mixes with $^{12}\text{CO}_2$ where $^{12}\text{C}$ is the most frequent stable isotope of carbon (it represents almost 99% of all naturally occurring carbon). Hence it is assimilated by plants and animals (through the normal carbon cycle). Although the $^{14}\text{C}$ of the terrestrial biosphere and oceans is constantly decaying, it is continually replenished from the atmosphere. Therefore the amount of $^{14}\text{C}$ stored in plant and animal tissue and in oceans remains approximately constant through time: an isotopic equilibrium has been reached.

When an organism dies, its metabolism and thus exchange with the environment stop. $^{14}\text{C}$ is no more replenished. It just decays at a constant rate. Thus, if the initial amount of $^{14}\text{C}$ ($N_0$) is known, the measure of the quantity that remains at the time of the discovery of the dead organism ($N$) gives its age ($t$): $N = N_0e^{-\lambda t}$ with $\lambda = \frac{\ln 2}{t_{1/2}}$.

Practically, the initial amount of $^{14}\text{C}$ is unknown. However the measure of the ratio $^{14}\text{C}/^{12}\text{C}$ of the sample compared to the one of the current biosphere gives the value of $N/N_0$ and thus the age of the death of the organism. In effect, the ratio $^{14}\text{C}/^{12}\text{C}$ of the biosphere is supposed to have remained constant through ages: that is the hypothesis of the isotopic equilibrium for C that was first considered as true until 1950 (after this date, nuclear experiments broke the equilibrium). Hence radiocarbon dates are expressed as ages BP (Before Present ie before 1950).

But actually this hypothesis is not reliable because of changes in the cosmic ray flux and in the carbon cycle (and it becomes more and more false with increasing age). To solve this problem, correlations where established with ages obtained by other methods as dendrochronology (that is based on the variations of size of the rings of trees) and a calibration curve for radiocarbon dates was thus built up.

Problems with radiocarbon dates occur due to calibration because of the variations of the curve. Due to "plateaux" (periods of constant radiocarbon age), an uncalibrated age BP -even if it is known with a good measure precision (as 50 years for example)- can produce a wide range of possible calibrated ages for the sample.

One method to solve partly this problem as well as uncertainties due to a lack of both accuracy and precision in the measure of ages is to take into account prior information such as stratigraphical context or succession (age should increase with depth) for calibrating a set of radiocarbon dates of a given sequence. This is the Bayesian analysis.
3.3.2 Analysis of radiocarbon chronologies: Bayesian approach

The data of an uncalibrated radiocarbon age BP is assumed to be represented by a normal distribution whose mean is the age and whose standard deviation is the measure uncertainty. Integrated against the calibration curve this gives an irregular more or less widespread distribution. For a given sequence, the set of plots that are obtained in that manner is called the prior (unconstrained) distribution.

In the Bayesian approach, using a software (OxCal) a constrained (posterior) distribution is generated by rejecting for each plot (ie for each initial date) the part of the delimited area that is incompatible with the other dates -because of age reversals in the stratigraphical sequence. Differences between prior and posterior distributions are expressed as an agreement index (for each initial age). If this index is lower than 60%, the date is rejected as inconsistent. With this method, a coherent chronology can be established for a sequence.

The new OxCal does not only use the stratigraphy of the sequence, it can also constrain the dates to fall within a deposition model assumed to follow a Poisson distribution whose parameter \(k\), chosen between 0 and 10, indicates the level of constraint (the higher \(k\), the more constrained).

I applied Bayesian analysis by using the new OxCal to establish precise chronologies extending over a few millennia during the Late Quaternary around the transition between Late-Glacial (15000-11000 cal years BP) and Holocene.

Figure 3: Chronology with tephra A and tephra B: depth vs. calibrated age BC with precise dates of the tephra added.
The first chronology was the one with tephra A and tephra B (see figure 3). Modelling the oldest part (Late Glacial) of the sequence was easy. Only three dates were rejected because of a poor agreement index. But in the sequence from early Holocene there were many reversals and incoherences. By rejecting before running OxCal the dates that were clearly false (due to a big reversal or because they were older than the age obtained for tephra B although the volcanic layer lied under the Holocene sequence), I could establish a chronology for that period with the new software. Then, I connected the two sequences and included the ages of the tephra (with indication of their depth) as "C-dates" (that did not need to be calibrated) within boundaries. The boundaries take into account a probable change in the celerity and regularity of deposition due to climate change. The result was a coherent chronology for the whole sequence.

The second spanned over the same period. It consisted in three records: two of England and one marine Adriatic record. As for the sequence with tephras, the two parts (Late Glacial) and Holocene had to be first worked out separately before being gathered. It was quite difficult to resolve the order because of the shifts between the different types of data (due to contamination): there were ages of macrofossils and ages of bulk sediments divided in a humic and in a humin fractions. Sometimes a whole category had to be rejected as apparently too old or too young. By using the new OxCal, the results that I obtained were very similar to those that had been already calculated with the old OxCal. As the new version constrains the dates to be included in a deposition model, this analysis proved again the reliability of the previous analysis.

4 Conclusion

In the first part of the project, the physical analyses have shown that a sample from Early Bronze Age contained very probably tin-slag. Indeed we could see in the XRF-spectrum a peak at 25 keV characteristic of tin; and the observation with the microscope in reflexive light showed shiny glassy grains with bubbles that may be traces of slag. These encouraging results have provided evidence for the interest of carrying out a bigger project about tin in this archaeological site.

The results that I obtained for the second part of the project with a new software that takes into account more prior informations have confirmed those obtained with an older version of the software, and reinforced the efficiency of tephrochronology and of Bayesian analysis to build accurate and precise chronologies of sequences of sediments with tephra layers.

This project was really interesting and was an illustration of how much different disciplines can complement each other. It shows how physics can be applied to archaeology and how essential scientific analyses are to a better understanding of human development particularly in prehistoric times.

As archaeology needs the combined works of different specialists in history and prehistory as much as science, researching in that field implies a very opened mind and a will of learning permanantly. Physicists or chemists in laboratories as the RLAHA in Oxford are more often specialized in a material (glass, bones or a particular metal for example) or a technique (radiocarbon or luminescence dating, tephrochronology,...) than a historical period. Therefore their projects can successively deal with various archaeological epochs and civilizations. By reading and speaking with human scientists they learn very much about the necessary historical background and their analyses provide key informations for the understanding of ancient societies. This work in collaboration (scientists do not only work in the laboratory, they often go to see archaeological sites where they speak with archaeologists, observe the geophysical environmental context, take the samples for analysis...) is very rich and efficient.
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Tephrochronology and radiocarbon dating of Quaternary sediments

Quaternary and dating methods


Tephrochronology


Age modelling and Bayesian analysis


The OxCal radiocarbon calibration software: http://c14.arch.ox.ac.uk/oxcal.php?group=O