PURBAWIDYA: **Jurnal Penelitian dan Pengembangan Arkeologi**
p-ISSN: 2252-3758, e-ISSN: 2528-3618 ■ Akreditasi Ristekdikti No. 21/E/KPT/2018
Vol. 7(2), November 2018, pp 167-174 ■ DOI: https://doi.org/10.24164/pw.v7i2.272

DENTAL PREPARATION PROCESS FOR THE ELECTRON SPINNING RESONANCE / U-SERIES DATING METHOD

Proses Preparasi Gigi Untuk Metode Penanggalan Electron Spinning Resonance/ U-Series

Anton Ferdianto
Balai Arkeologi Jawa Barat
Jalan Raya Cinunuk KM 17 Cileunyi, Bandung
E-mail: antonferdianto18@gmail.com

Naskah diterima 15 September 2018 – Revisi terakhir 29 Otober 2018
Disetujui terbit 12 November 2018 – Tersedia secara online 30 November 2018

Abstrak

Fosil tulang dan gigi merupakan temuan yang biasanya ditemukan dalam penelitian arkeologi. Di beberapa tempat temuan tulang yang memiliki jejak potong oleh aktifitas manusia memberikan informasi yang sahih akan keberadaan manusia, oleh karena itu tulang dan gigi merupakan objek yang sangat tepat untuk dilakukan pengujian umur pada lapisan arkeologi. Pada penanggalan geokronologi, penggabungan dua metode seperti ESR/U-series semakin sering digunakan tiga dekade belakangan ini. Metode ini dapat digunakan di situs yang memiliki konteks geologi berbeda – beda (fluvial, lacustrine atau karstic) dalam rentang waktu 300.000 – 20.000.000 tahun yang lalu. Selain itu metode ini dapat diaplikasikan tidak hanya untuk gigi fauna namun dapat diaplikasikan juga untuk gigi hominid. Akan tetapi untuk menggunakan metode ini diperlukan beberapa tahapan yang harus dilakukan. Dalam artikel ini akan dijelaskan mengenai tahapan yang harus dilakukan sebelum sampel tersebut siap untuk dihitung umurnya, mengingat masih sedikit tulisan yang menyinggung mengenai proses persiapan tersebut secara terperinci.

Keywords: penanggalan, Electron spinning resonance, preparasi gigi.

Abstract

Fossil bones and teeth are findings that are usually found in archeological research. In some places where bone findings have traces of human activities, provide valid information about human existence, therefore bones and teeth are very appropriate objects for age testing at the archeological layer. In geochronological dating, the merger of two methods such as the ESR and U-series is increasingly being used in the past three decades for dating. This method can be used on sites that have different geological contexts (fluvial, lacustrine or karstic) in the span of 300.000 – 20.000.000 years ago. Besides, this method can be applied not only for fauna teeth but can also be applied to hominid teeth. However, to use this method several steps must be taken. In this article we will explain the steps that must be taken before the sample is ready to be calculated for its age, considering that there are still a few writings that pertain to the preparation process in detail.

Keywords: dating, electron spinning resonance, dental preparation.

INTRODUCTION

Bones and teeth usually are very common in archeological findings. At numerous sites these remains, such as bones bearing cut marks from human activities, may provide unequivocal evidence for human occupation. Thus bones and teeth provide the ideal targets for directly dating of archeological levels.

Mammalian tooth consists of three tissues: enamel, dentine, and cement. Enamel contains ~96% (by weight) calcium phosphate (hydroxyapatite), ~3% water, and ~1% organic matter; dentine and cement contain 70-75% calcium phosphate, ~20% organic matters, and 5-10% water (Driessens and Verbeeck, 1990). After being deposit, the teeth will be altered by the fossilization processes of biological decay of organic constituents and inorganic transformations of crystalline, leading to profound changes of chemistry and structure of the buried teeth (Schwarcz, 2000; Kohn, 2008). The tissues of dentine and cement have close similarities to bone in their fossilization response (Kohn, M. J. and Law, J. M., 2006), while enamel is much more resistant to alteration because of less organic content and larger crystal size (Kohn, M. J., Schoeninger, M. J., Barker, W. W., 1999)

Combined ESR/U-series dating of teeth has been increasingly used in geochronological research over the past two decades. Results prove that it can be potentially applied to different geological contexts sites (fluvial/lacustrine or karstic environments) and an interesting dating method for the studies of the Pleistocene human migrations. The teeth preperation protocol had been writen in Shao (2011) disertation on his protocol diagram, but he did not discribe it very clear in each step of the protokol. Therefore the aim of this article is to explain how to prepare the teeth samples for ESR and U/series dating method in each protocol step. These processes indeed require a strict and standard operation procedure (SOP) to have a good result for geochronologist who want to work on ESR/U-series dating method.

SITES AND SAMPLES

There's six teeth sample that had been use in this laboratory work. Two samples are from Matar (Sangiran, Indonesia) provide by Prof. Truman Simandjuntak from the Center of Archaeology Research in Indonesia and four teeth samples are from Cueva del Angel (Lucena, Spain) provide by Prof. Christophe Falguères from MNHN.

Matar site are located in Ngelo Village, Bojonegoro Regency, East Java. This site is a part on the Bengawan Solo river system that streched for about 600 km. This site are are being excavated by Pusat Penelitian Arkeologi Nasional (Puslit Arkenas) since 2012 until now and unearthed huge amount of archaeological and paleontological deposit. Several research in quartenary geology revealed that the teraces are containing deposit since Pleistocen period.

From the last excavation that had been done in Matar site, it was revealed more than 3.000 object, including faunal remains, and stone artefact. From the stone artefact we could see that there was a similarity with the Paleolithic artefact

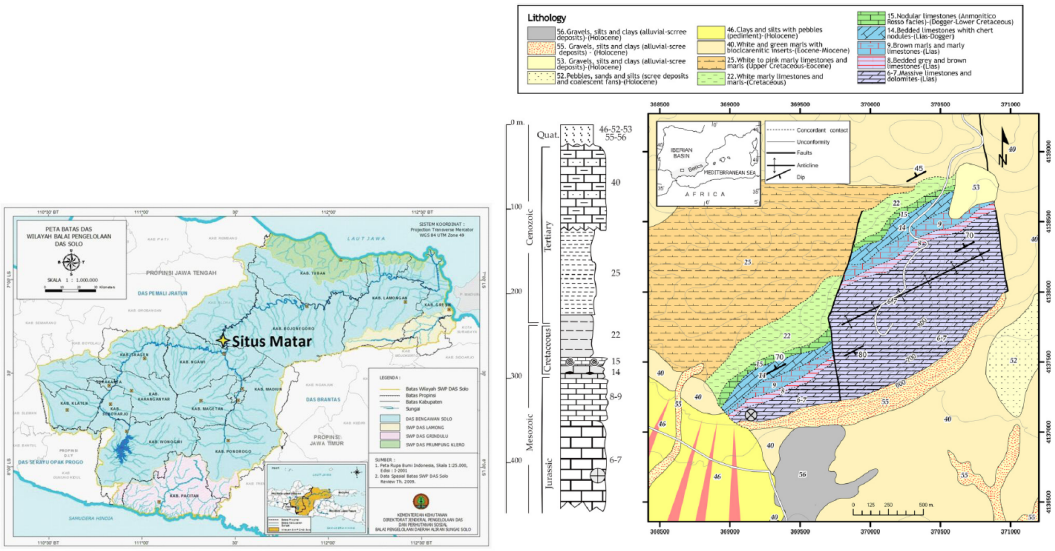

Figure 1. Geological map of Matar Sites (left) (Nikko. A, et. al. 2013), Geological map and litostratigraphy of Cueva del Angel sites (right) (Barrozo Ruiz. et. al. 2011)

that had been found in Sangiran, Baksoko and Ngandong in this case "bola batu" stone artefact. This appearance showed us existance of the Acheulian (Clark's African mode 2 technology) which came from Africa about 1,6 million years ago. from the faunal remains gives us an information that there was a similarity with the Ngandong fauna.

The Cueva del Angel archaeological site is an open-air sedimentary sequence, remnant of a collapsed cave and part of a karst complex. The faunal assemblage dominated by Equus ferus, large bovids and cervids has been subjected to intense anthropic actions reflecting selective predation. The fauna may be correlated with European faunistic associations of the end of the Middle Pleistocene to the beginning of the Upper Pleistocene. The Cueva del Angel lithic assemblage (dominated by non-modified flakes and abundant retouched tools with the presence of 46 handaxes) appears to fit well within the regional diversity of a well developed non-Levallois final Acheulean industry (Barroso Ruíz et al., 2011).

CA1301 CA1307 CA1306

CA 1309

Figure 2. Teeth samples from Cueva del Angel (Source: Document Ferdianto, 2014)

48 AY81

Figure 3. Teeth samples from Matar (Source: Document Ferdianto, 2014)

METHODS AND SAMPLE PREPARATION

In the field, the samples (mammal teeth) and the surrounding sediments are collected, and some measurements of the in-situ γ-dose rate are realized in the immediate environment of collected teeth. The teeth of the Equid, Bovid, Cervid, and Rhinocerose taxa are the

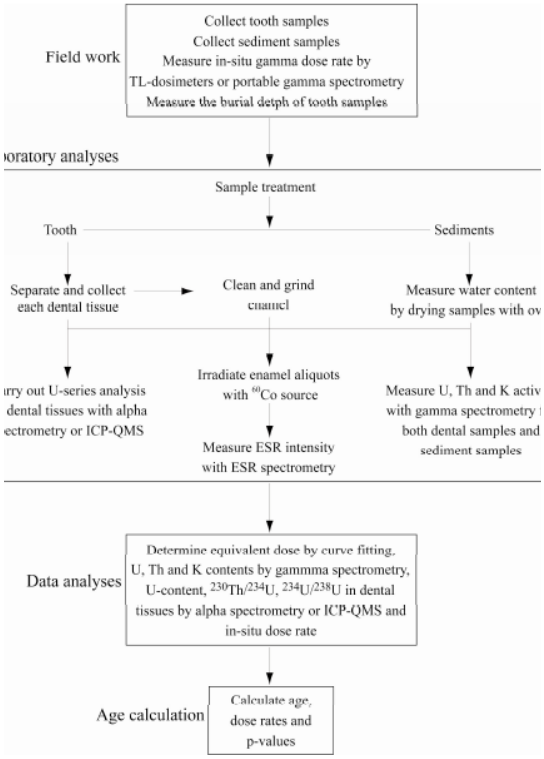

Figure 4. Strategy of combined ESR and U-series dating of tooth enamel for the present work, in Shao (2011) modified from Bahain et al. (2002).

most favorable samples for ESR dating because of their big size and thick enamel layers eventhough that smaller sample such as i.e. hominid teeth still possible. If possible, we preferentially choose the teeth that can provide a piece of flat and thick (> 100 μm) enamel with mass > 1g as dating samples. The sediments, in which the tooth samples were preserved, are required for assessment of local dose rate, especially for the β- and γ-rays contributions. In the field the sediments (minimum 100 – 200 g) are collected in radium of about 30 cm around the tooth sample, considering the effective range of γ-rays emitted by U, Th and K. If the burial condition is too complicated to find representative sediments (heterogeneous stratigraphy or lumpy sites), it is very important to measure the in-situ dose rate with thermoluminescence (TL) dosimeters (Valladas, 1982) or portable γ-ray spectrometry (Mercier and Falgueres, 2007). Additionally, we need to measure the burial depth of the tooth sample to evaluate the cosmic dose rate (Prescott, J. R., Hutton, 1994).

In laboratory, the first step is to prepare the collected samples, making them suitable for ESR and U series analysis. For tooth, we separate, clean and grind mechanically enamel, then make aliquots for artificial irradiation and later for ESR signal measurement. For sediments, we measure the water content by drying the samples with oven. The separated dentine, cement, rest of the enamel, and dried sediments are then measured by γ-ray spectrometry to test their contents of U, Th and K. Then, we carry out U-series isotopic analyses on the dental samples.

After finishing field and lab work a lot of primary data are obtained. In order to extract the necessary information required for combined ESR and U-series age estimation, we need to perform many data analyses.

SAMPLE TREATMENT

Registering the Sample

The first step that we need in laboratory analysis is to register the entire sample in the ESR laboratory database. By registering the sample we could be able to trace back the progress and the stage of the sample during the preparation and analyzing. This data base also provides the information of how many samples that had been analyzed in the lab, when it is start being prepared and analyzed. Also all the information of the sample such as sites, contexts and its condition. Taking a picture is necessary to be done regarding the next step it will be more destructive. In some case if the teeth are small or fracture in small pieces you need to use all the part of the tooth to have enough samples for measuring in ESR/ U-series. So, by taking a picture to the sample from each side, provide us the exact form and size of the sample.

Separating the Enamel, Dentin and Cement

Mammalian tooth is composed of three dental tissues: enamel, dentine and cement, which are arranged in a layered structure. The tooth enamel mainly consists of crystallized hydroxyapatite, and is used as the natural dosimeter accumulating the dose given by the adjoining dentine, cement, sediments and the injected cosmic rays. The first goal of tooth sample preparation is to obtain pure enamel samples free of residual dentine and cement, because they contain much more organic components and U than enamel. Secondly, the dentine and cement adhered to the enamel also needs to be separated from each other for U-series analysis, because they usually have different U contents and different U-uptake behaviors with enamel, and thus they contribute different dose rates to tooth enamel.

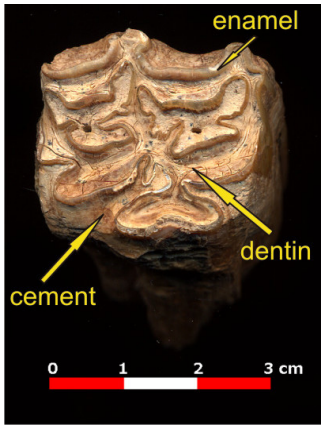

Figure 5. Patterns of enamel, dentine and cementum (Source: Document Ferdianto, 2014)

To facilitate the tissue separation, the tooth was firstly cut into two halves or crushed into a few large pieces. Then the dentine and cement were split from enamel, mechanically. The dentine and cement were collected separately for following γ-spectrometry measurement and U series isotopic analysis.

Enamel Reduction

The enamel pieces were precisely measured with a micrometer for thickness, and further cleaned by removing off a layer ~50 μm thick from both inside and outside with dental drill.

The removal of the enamel surfaces is realized both to clean enamel layer of any contamination by dentine, cement or sediment and to eliminate external α-radiation influence, which has effective range of 20 - 40 μm. Such cleaning treatment meanwhile reduces

the β-radiation coming from the attached dental tissues or sediments, because β particles have effective range of about 2 mm, comparable to enamel thickness. The removed enamel thickness from both sides need to be precisely measured for evaluating the β- dose attenuation caused by the removal of enamel surfaces.

Grinding and Sieving

Finally, the cleaned enamel pieces were ground with mortar and pestle. The powdered enamel between 100 and 200 μm was weighted and put it in the different container. But if we gain less than one grams of powdered enamel from both size due to lack of enamel sample, we should put it in the same container. But this condition will make the measurement have to be done one by one (ESR first then the U-series) because we were using the same sample and also in the sample preparation, it should be avoided to heat the enamel by mechanical operations, because temperature increase may generate interfering ESR signals in enamel.

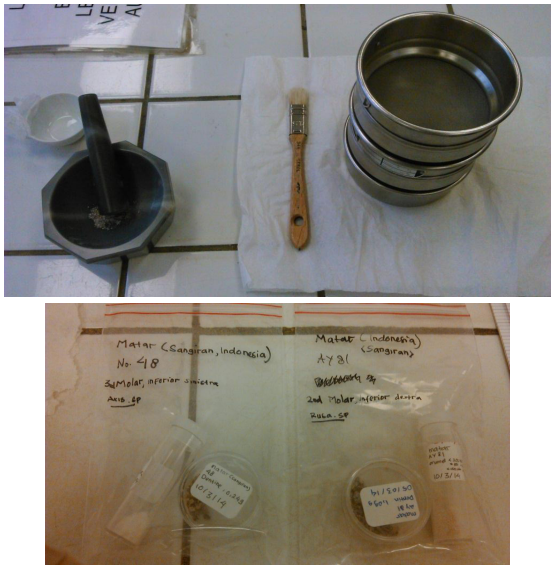

Figure 6. Grinding, sieving of the enamel (Source: Document Ferdianto, 2014)

REMARKS AND DISCUSSION

The teeth preparation for ESR/U-series is very important step and needs to be very precise. You need to be very careful in each step of the preparation and make sure that everything is according to the protocol. Make sure that there's no contamination during the preparation. This contamination or mistake on the preparation could lead to the wrong result on the age estimation.

Figure 7. Seperation of cementum, enamel and dentin (Source: Document Ferdianto, 2014).

From the entire sample that had been prepared, we could see that sample from Matar, are very difficult to prepare, because the sample are quite small and very fragile due to the condition of the sample itself. It's very different with the

sample from Cueva del Angel, in terms of size and condition that much better way so, is quite easy to prepare.

Continued advancement in technology and scientific instrumentation make it possible to analyze prehistory collections to answer a wide range of questions especially in estimating the age of the object. However, some of the proposed procedure and methodology are invasive and pose great challenges into issues of balancing conservation and analytical procedures.

BILIOGRAPHY

Barroso Ruíz, C., Botella Ortega, D., Caparrós, M., Moigne, A. M., Celiberti, V., Testu, A., Barsky, D., Notter, O., Riquelme Cantal, J. A., Rodríguez, M. P., Carretero León, M. I., Monge Gómez, G., Khatib, S., Saos, T., Gregoire, S., Bailón, S., García Solano, J. A., Cabral Mesa, A. L., Djerrab, A., George Hedley, I., Abdessadok, S., Batalla LLasat, G., Astier, N., Bertin, L., Boulbes, N., Cauche, D., Filoux, A., Hanquet, C., Milizia, C., Moutoussamy, J., Rossoni, E., Verdú Bermejo, L. and de Lumley, H. (2011) 'The Cueva del Angel (Lucena, Spain): An Acheulean hunters habitat in the South of the Iberian Peninsula', Quaternary International. Elsevier Ltd and INQUA, 243(1), pp. 105–126. doi: 10.1016/j.quaint.2011.02.021.

Driessens, F. C. M. and Verbeeck, R. M. H. (1990) Biominerals. VRC Press.

Kohn, M. J., Schoeninger, M. J., Barker, W. W. (1999) 'Altered states: effects of diagenesis on fossil tooth chemistry', Geochimica et Cosmochimica Acta, 63(18), pp. 2737–2747.

Kohn, M. J. and Law, J. M. (2006) 'Stable isotope chemistry of fossil bone as a new paleoclimate indicator', Geochimica et Cosmochimica Acta, 70(4), pp. 931–946.

Kohn, M. J. (2008) 'Models of diffusion-limited uptake of trace elements in fossils and rates of fossilization', Geochimica et Cosmochimica Acta, 72(15), pp. 3758–3770.

Nikko, SD, A., Truman Simandjuntak., M. R. Fauzi., M. M. Ansyori., D. Prastiningtyas., Wilandari., Sugiharno., Wardoyo., 2013, Laporan Kajian Potensi Budaya DAS Bengawan Solo, Penelitian Situs Matar, Desa Ngelo, Margo Mulyo, Bojonegoro, 11-69, BPSMP Sangiran.

Prescott, J. R., Hutton, J. T. (1994) 'Cosmic ray contributions to dose rates for luminescence and ESR dating: large depths and long-term time variations', Radiation Measurements, 23, pp. 497–500.

Schwarcz, H. P. (2000) 'Dating bones and teeth: the beautiful and the dangerous', in P. V. Tobia, M. A. Raath, J. Moggi-Cecci, and G. A. Doyle (ed.) Humanity from African Naissance to Coming Millennia. Florence: Firenze university Press, pp. 249–256.

Shao, Qingfeng, 2011, Combined ESR/U-series Dating of Fossil Teeth from Middle Pleistocene Sites in Northern Europe and Mediterranean Area: Contributing to the Chronology of the Acheulian Settlement of Europe, Doctoral Thesis, MNHN. Paris, (not publish).

Valladas, G. (1982) 'Mesure de la dose gamma annuelle de l'environnement d'un site archéologique par un dosimètre TL', PACT, 6, pp. 77–85.