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ESR dating of quartz extracted from Quaternary sediments
application to fluvial terraces system of northern France [Datation
par résonance paramagnétique électronique (RPE) de quartz
fluviatiles quaternaires: application aux systèmes de terrasses du
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Abstract

Since the 1980's, the improvement of the Electron Spin Resonance (ESR) method and its application to the dating of bleached quartz extracted from sediments currently allow to provide geochronological data on Pleistocene fluvial deposits and associated archaeological sites. The ESR dating of quartz may hence improve our knowledge on Quaternary chronostratigraphy, especially in case of azoic deposits, when classical methods can't be applied. The ESR method is based on the behaviour of aluminium-center in quartz exposed to solar light and ionizing radiations. This center can be optically bleached during the fluvial transport but, as this bleaching is incomplete, a residual dose must be determined and used for the age calculation. After a short description of the general principle of the ESR dating, the methodological basis and analytical procedures corresponding to the quartz are detailed, then the potential of the ESR method is demonstrated through the presentation of the results obtained from the study of the fluvial terraces of the Creuse and Yonne valleys (France).

Résumé

Depuis les années 1980, le développement de la méthode de datation par Résonance Paramagnétique Electronique (RPE) de quartz blanchis extraits de sédiments, a permis d'obtenir de nombreux âges pour des dépôts fluviaux fossiles et pour les sites archéologiques qu'ils contiennent. Cette méthode a ainsi permis de développer notre connaissance de la chronostratigraphie des systèmes fluviaux pendant toute la période du Quaternaire, particulièrement dans les cas où il s'agit de dépôts azoïques, quand des méthodes plus " classiques " ne sont pas applicables. La datation par RPE sur quartz est basée sur les réactions du centre aluminium du quartz face à la lumière solaire et aux rayonnements ionisants. Ce centre peut être optiquement blanchi pendant la phase de transport des grains, mais ce blanchiment est incomplet. Une dose résiduelle doit être déterminée pour pouvoir calculer l'âge de l'échantillon. Après une courte description de la méthode RPE, les bases méthodologiques et analytiques correspondant au cas particulier du quartz seront détaillées, puis le potentiel de cette méthode sera démontré à travers la présentation des résultats obtenus pour les vallées de la Creuse et de l'Yonne.

ESR DATING OF QUARTZ EXTRACTED FROM QUATERNARY SEDIMENTS APPLICATION TO FLUVIAL TERRACES SYSTEM OF NORTHERN FRANCE

■
Pierre VOINCHET*, Jean-Jacques BAHAIN*, Christophe FALGUÈRES*, Michel LAURENT*, Jean-Michel DOLO**, Jackie DESPRIÈRE*, Robert GAGEONNET* & Christine CHAUSSÉ***

ABSTRACT

Since the 1980's, the improvement of the Electron Spin Resonance (ESR) method and its application to the dating of bleached quartz extracted from sediments currently allow to provide geochronological data on Pleistocene fluvial deposits and associated archaeological sites. The ESR dating of quartz may hence improve our knowledge on Quaternary chronostratigraphy, especially in case of azoic deposits, when classical methods can't be applied. The ESR method is based on the behaviour of aluminium-center in quartz exposed to solar light and ionizing radiations. This center can be optically bleached during the fluvial transport but, as this bleaching is incomplete, a residual dose must be determined and used for the age calculation. After a short description of the general principle of the ESR dating, the methodological basis and analytical procedures corresponding to the quartz are detailed, then the potential of the ESR method is demonstrated through the presentation of the results obtained from the study of the fluvial terraces of the Creuse and Yonne valleys (France).

Key-words: Electron spin resonance (ESR), quartz, optical bleaching, Pleistocene fluvial deposits.

RÉSUMÉ

DATATION PAR RÉSONANCE PARAMAGNÉTIQUE ÉLECTRONIQUE (RPE) DE QUARTZ FLUVIATILES QUATERNAIRES : APPLICATION AUX SYSTÈMES DE TERRASSES DU NORD DE LA FRANCE

Depuis les années 1980, le développement de la méthode de datation par Résonance Paramagnétique Electronique (RPE) de quartz blanchis extraits de sédiments, a permis d'obtenir de nombreux âges pour des dépôts fluviaux fossiles et pour les sites archéologiques qu'ils contiennent. Cette méthode a ainsi permis de développer notre connaissance de la chronostratigraphie des systèmes fluviaux pendant toute la période du Quaternaire, particulièrement dans les cas où il s'agit de dépôts azoïques, quand des méthodes plus "classiques" ne sont pas applicables. La datation par RPE sur quartz est basée sur les réactions du centre aluminium du quartz face à la lumière solaire et aux rayonnements ionisants. Ce centre peut être optiquement blanchi pendant la phase de transport des grains, mais ce blanchiment est incomplet. Une dose résiduelle doit être déterminée pour pouvoir calculer l'âge de l'échantillon. Après une courte description de la méthode RPE, les bases méthodologiques et analytiques correspondant au cas particulier du quartz seront détaillées, puis le potentiel de cette méthode sera démontré à travers la présentation des résultats obtenus pour les vallées de la Creuse et de l'Yonne.

Mots-clefs : Résonance Paramagnétique Electronique (RPE), quartz, blanchiment optique, dépôts fluviaux pléistocènes.

INTRODUCTION

The Plio-Pleistocene fluvial terraces systems represent a great interest both for geologists and archaeologists, because they have recorded tectonic uplift, climatic variations and human occupations. The dating of such alluvial

deposits allows to elaborate regional chronostratigraphical frameworks in which the geological, palaeoenvironmental and archaeological evolutions can be replaced.

Since the end of the 1980's, electron spin resonance (ESR) dating of bleached quartz extracted from sediments provides geochronological data on Pleistocene

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sedimentary deposits. The method can be applied on aeolian, littoral and fluvial sediments in which quartz have been exposed to solar light during the transport phase, before the final burial. After a brief survey of the ESR method and of its application to the dating of bleached quartz, this paper illustrates the potential of ESR method through some examples concerning Pleistocene fluvial systems of Northern France.

ESR DATING METHOD

The ESR method is a classical way of analysis of the solid state, used in chemistry, cristallography and physics. Its geochronological application was proposed at the end of the 1960's, and since then the method was mainly used to date geological or archaeological remains as tooth enamel, marine or continental carbonates and quartz. Reviews on ESR dating can be found in several synthesis papers or books (for example, Grün, 1989; Ikeya, 1993; Rink, 1997; Falguères and Bahain, 2002).

Among the available materials, the potential of quartz is important for dating, because the presence of various paramagnetic ESR centers in its structure allows the dating of different types of geological or archaeological events: fault movements (Ikeya *et al.*, 1982; Toyoda and

Szwarcz, 1997), thermal annealing (Yokoyama *et al.*, 1985; Toyoda and Ikeya, 1989, 1991, 1994; Miallier *et al.*, 1994; Imaï *et al.*, 1985) or sediment deposits (Yokoyama *et al.*, 1985; Falguères *et al.*, 1988; Laurent *et al.*, 1998). Then the calculated age does not correspond to the mineral growth, but to a major post-crystallization event, which leads to a zeroing of the ESR centers by pressure, heating or optical bleaching. It becomes hence possible to date volcanic or tectonic phases and fluvial, aeolian or littoral deposits.

As for the thermoluminescence and optoluminescence methods, the ESR dating method is based on the use of geological or archaeological materials as dosimeters, which have recorded the total dose of radiations received by the sample during its history.

Natural radiations emitted by radionuclides as uranium, thorium and their daughters, and potassium, produce free electrons. A part of these electrons may be trapped by impurities or lattice defects of the minerals and the total amount of trapped electrons in a geological or archaeological material is then proportional to the age of the sample :

$$TD = \int_0^T da(t).dt \quad (\text{formula 1})$$

where TD is the *total dose* of radiations received by the sample, d_a is the *annual dose rate* of natural radiations and T the age of the sample. The calculation of an ESR age needs therefore the determination of both TD and d_a parameters.

The total dose TD is proportional to the trapped electrons concentration in the sample and so to the ESR signal intensity. This dose is determined by the additive dose method. Several aliquots of the sample are submitted to various gamma-ray doses (*multiple aliquots method*) in order to obtain a dose-response curve that allows determine the total dose value by extrapolation using a simple exponential function with the least-square method (Apers *et al.*, 1981; Yokoyama *et al.*, 1985) (fig. 1):

The annual dose rate, d_a , is the sum of both internal and external dose rate contributions, related to α , β , γ and cosmic radiations emitted from the environment and from the datable material itself.

PARTICULARITIES OF ESR DATING OF OPTICALLY BLEACHED QUARTZ

As said before, the ESR dating of alluvial quartz is based on the hypothesis of an ESR-traps zeroing by sunlight during the aqueous transport phase in a river prior to the sediment burial. The calculated age does not correspond to the crystallization, but to the time elapsed since the deposit, called archaeological period in fig. 2.

Effectively, some of the different impurity ESR-centers identified in quartz (see Weil, 1984) are photo-sensitive, especially titanium (Ti) - and aluminium (Al) - centers, which are useful for geochronological analyses (Yokoyama *et al.*, 1985). When a photo-sensitive center is optically excited by light, the trapped electrons are released and the corresponding ESR signal intensity decreases. This phenomenon is called *optical bleaching*.

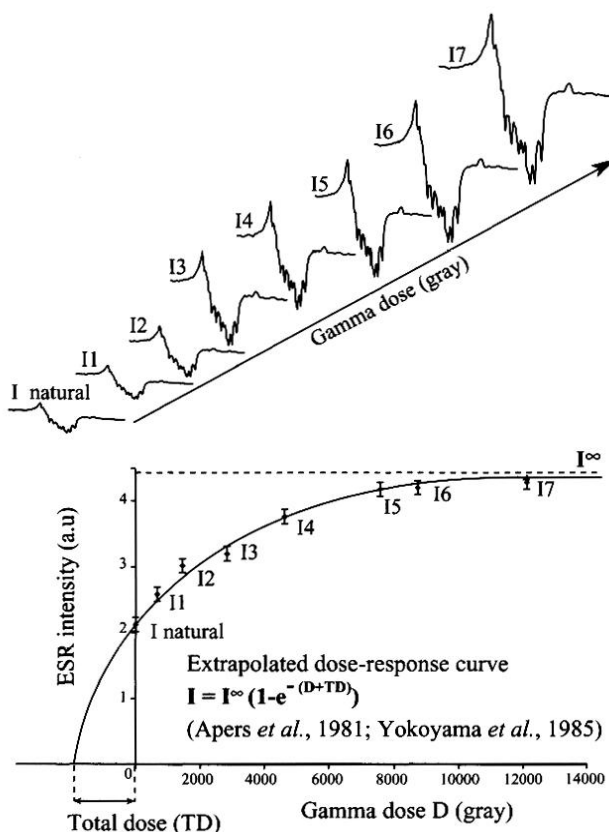


Fig. 1: Evolution of the ESR intensity for the Aluminium-center of quartz versus increasing doses of radiations and corresponding dose-response curve.

The mathematical function used for the total-dose extrapolation is a single exponential function, according with Apers *et al.* (1981) and Yokoyama *et al.* (1985).

Fig. 1 : Evolution de l'intensité du signal RPE du centre Aluminium du quartz en fonction de doses croissantes de radiations et courbe de réponse à la dose correspondante.

*La fonction mathématique utilisée pour l'extrapolation de la dose totale est une fonction exponentielle simple, en accord avec Apers *et al.* (1981) et Yokoyama *et al.* (1985).*

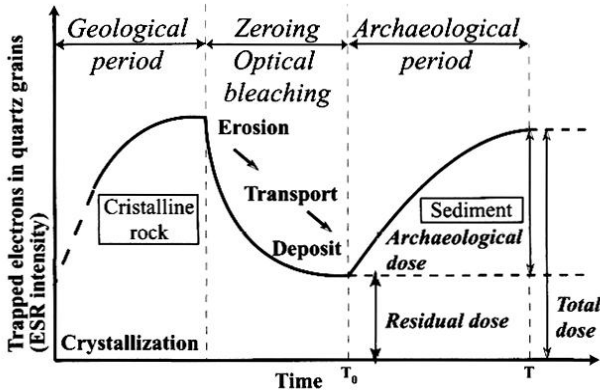


Fig. 2: Schematic history of the evolution of the ESR Al-center intensity in quartz extracted from fluvial sediments.

The zeroing phenomenon used for the ESR dating corresponds to an incomplete optical bleaching of the traps during the transport phase of the sediment by the river. After the deposit and the burial of the sediment, the natural irradiation of the sample leads to a growth of the ESR signal intensity. The determination of the "archaeological dose", which corresponds to the amount of traps filled since the burial, necessitates the knowledge of both total and residual (non-bleachable component) doses.

Fig. 2 : Histoire schématique de l'évolution de l'intensité RPE du centre Aluminium dans des quartz extraits de sédiments fluviaux.

Le phénomène de remise à zéro utilisé pour la datation RPE correspond à un blanchiment optique incomplet des pièges durant la phase de transport des sédiments par la rivière. Après le dépôt et l'enfouissement du sédiment, l'irradiation naturelle de l'échantillon entraîne une augmentation de l'intensité du signal RPE. La détermination de la "dose archéologique", qui correspond à la quantité de pièges remplis depuis l'enfouissement, nécessite la connaissance à la fois de la dose totale et de la dose résiduelle (composante ne pouvant être blanchie).

However, for the Al-center, the bleaching is incomplete. Only a part of trapped electrons is released, even after a long-time light-exposure. In fact, the ESR-intensity decreases until a plateau-value corresponding to the electrons, which cannot be «extracted» by the energy provided by light. The "residual dose" hence obtained must be subtracted from the total dose in order to obtain the dose really received by the sample since its burial (archaeological dose), used for age determination (fig. 2). Therefore, for each ESR dating analyses, it is crucial to determine the residual dose of each sample.

Various procedures were proposed for this determination. First, Yokoyama *et al.* (1985) used the ESR intensity of recent sediment, considered as maximally bleached. The main difficulty of this way concern the choice of this sediment, which needs to have the same general characteristics of the fossil one. More recently, the study of the bleaching curves of fossil samples has permitted to propose mathematical functions describing the decrease of Al-center intensity versus the light-exposure time (Walther et Zilles, 1994; Toyoda *et al.*, 2000; Voinchet *et al.*, 2003). In practice, the observed decreasing curve can be expressed by a double exponential function and the maximal bleaching is done by exposing the quartz samples under an ultra-violet light during a six months equivalent-time (fig. 3a) (Voinchet *et al.*, 2003). This period could appear to be long, but a study performed on present fluvial deposits has permitted to confirm that the maximal bleaching was really reached (fig. 3-b). This validates the use of ESR dating of alluvial deposits.

The sediments are sampled in cleaned sections in order to minimize parasite bleaching. In the lab, a 100–200 μm fraction is selected by water sieving. Quartz is then separated using a chemical and physical protocol already detailed in the literature (Yokoyama *et al.*, 1985; Laurent *et al.*, 1994; Voinchet, 2002).

After this treatment, the sample is split in several aliquots used for the residual dose and total dose determinations. Irradiation is performed using a panoramic ^{60}Co source (Dolo *et al.*, 1996) emitting a 1.25 MeV γ -ray with a dose rate of 200 Gy/h. Generally, height aliquots are submitted to doses ranging between 500 and 30,000 Gy. The determination of the residual intensity is realized with the experimental device described in Voinchet *et al.* (2003). Three halogen lamps and one UV lamp imitating the solar spectrum were used to illuminate the quartz grains. The light intensity then received by each aliquot ranges between 3.2 and 3.4 10^5 Lux. The maximal bleaching is attempted after an exposure of around 1000 hours. The maximal bleaching percentage (Bl. %) is then calculated using the following formula:

$$\text{Bl. \%} = (I_{\text{nat}} - I_{\text{bl}}) / I_{\text{nat}} \quad (\text{formula 2})$$

where I_{nat} and I_{bl} correspond respectively to the natural ESR-signal intensity and to the maximally bleached one.

ESR measurements of the Al-center intensity at the temperature of liquid nitrogen ($\approx 90\text{K}$) were performed with a Brüker EMX spectrometer using the experimental conditions proposed by Yokoyama *et al.* (1985). The Al-signal intensity is measured between the top of the first peak $g=2,018$ and the bottom of the 16th peak $g=2,002$ of the part of the main hyperfine structure.

The annual dose rate is calculated from the radionuclides activities obtained by gamma-ray spectrometry measurements. Alpha and beta attenuations in quartz

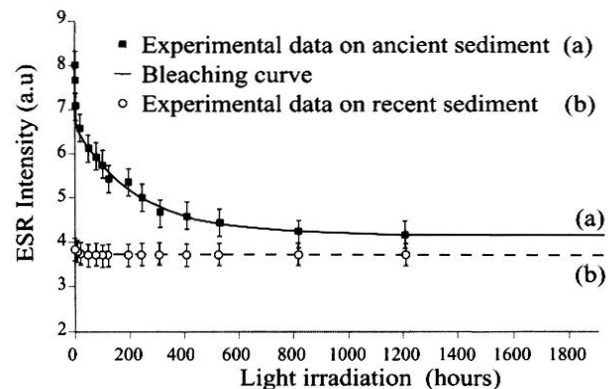


Fig. 3: Optical bleaching curves of the ESR Al-center for quartz extracted from fossil (a) and recent (b) sediments.

The maximal bleaching of the fossil sample is obtained after a 1000 hours exposure to the experimental device described in Voinchet *et al.* (2003). It corresponds to an equivalent six months exposure to the solar light.

In the other hand, the Al-center of quartz issued from recent sediment is practically maximally bleached. It validates the hypothesis of initial optimal bleaching in natural conditions.

Fig. 3 : Courbes de blanchiment optique du centre RPE aluminium pour des quartz extraits de sédiments fossiles (a) et actuels (b).

Le blanchiment maximal de l'échantillon fossile est obtenu après une exposition de 1000 heures au dispositif expérimental décrit dans Voinchet *et al.* (2003). Cela correspond à une exposition à la lumière solaire d'environ six mois.

were estimated from the tables of Bell (1980) and Mejdahl (1979) and the alpha dose is calculated using an alpha-ray efficiency-correcting factor (k-value) of 0.15 (Laurent *et al.*, 1998). The cosmic dose depends on the altitude, latitude and on the sediment thickness above the sample (Yokoyama *et al.*, 1982).

ESR DATING OF FLUVIAL SYSTEMS OF NORTHERN FRANCE: SOME EXAMPLES

The application of ESR dating of bleached quartz has been successfully applied on Plio-Pleistocene marine sediments in France and in Italy (Yokoyama *et al.*, 1985; Gagnepain *et al.*, 1998). During the 1980's, the ESR study of the Somme River system sediments was performed by Laurent *et al.* (1994, 1998) and permits, in relation with combined ESR/U-series, magnetostratigraphical, geological and palaeoenvironmental data, to elaborate a complete chronological framework for this valley (Antoine *et al.*, 2000).

Since 1997, ESR dating of bleached quartz was systematically applied on sediments carried out from several fluvial systems of the northern part of France including the Loir River (Despriée *et al.*, 2003), the Creuse River (Despriée *et al.*, 2004, this issue), and the Yonne River systems (Chaussé *et al.*, 2004, this issue). Some results obtained during this study (Voinchet, 2002) illustrate well the potential and the present limits of the ESR method and will be detailed now. For each studied site, analytical data tables (archaeological and annual doses, water content in sediment, maximal bleaching percentages and ages) are presented on the respective illustrations.

DATING OF THE LOWER TERRACES OF THE YONNE RIVER SYSTEM AND OF THE PALAEO-LITHIC SITES OF SOUCY

The Yonne River is a tributary of the Seine River, which flows through the southern part of the Paris Basin in the Burgundy. Since several years, new geological and archaeological researches directed by Christine Chaussé, Vincent Lhomme and Nelly Connet have allowed to better know the stepping of the fluvial terraces and the palaeolithic settlement of the valley. In the Yonne River system, the most important site is located at Soucy, where six different Lower Palaeolithic localities with palaeontological and archaeological remains have been discovered on the same fossil fluvial terrace of the Yonne River, at + 20 m of relative altitude above the present river bedrock (Lhomme *et al.*, 1997, 2000; Chaussé *et al.*, 2000). Three other alluvial sheets are intercalated between the Soucy Sheet and the modern floodplain, respectively at +6 m, +10 m and +15 m (fig. 4) (Chaussé *et al.*, 2004).

The Soucy sites gave the opportunity to compare ESR results derived from the dating of bleached quartz with those obtained by combined ESR/U-series methods (US model) on tooth enamel (see Grün *et al.*, 1988). Four sediments of the fine fluvial Soucy sequence and twelve horse teeth have been analysed (fig. 4) (Voinchet, 2002). The whole ESR data are homogeneous and assign a quadratic mean age of 356 ± 14 ka to the Soucy Sheet. These

results are completed by further analyses realized on samples carried out from different localities of the same sheet, Gron and Michery. For this last sample, a contamination of the sediment by material removed from an older alluvial deposit is suspected to explain the ancient age.

Respecting the geological evidences, the formation of the Soucy fluvial terrace is contemporaneous with the oceanic isotopic stage (OIS) 10 and these data places the human occupations during the OIS 9. The ESR results are coherent with geological and palaeoenvironmental data (Lhomme *et al.*, 1997, 2000; Chaussé *et al.*, 2000). The ages obtained on the lowest terraces permit to propose a correlation of the Misy (+15 m) and Evry (+10 m) Sheets with the OIS 8 and 7. The Sens-Chambertrand (+5 m) and Gron (+3 m) Sheets could correspond to the OIS 6 to 3. Geological data (Chaussé *et al.*, 2004) and archaeological discoveries, in particular the discovery of Mousterian Quina and Aurignacian levels in the fluvial sediments of the Gron Sheet, permit here to better calibrate the ESR results. In fact, the sensibility of the Al-center used for the dating seems to be too poor to allow a better discrimination of the alluvial formations younger than 200 ka. Methodological works are still in progress to study the possible use of the ESR titanium-center, very much sensible than the Al-center, for the dating of such levels.

RESULTS OBTAINED ON THE CREUSE RIVER SYSTEM IN THE "MASSIF CENTRAL" SECTOR

The Creuse River is a tributary of the Loire catchment basin, in the central part of France, on the northern edge of the Massif Central and the southern and western parts of the Paris Basin. Recent researches have shown the impact of the recent tectonics and of the nature of the bedrock on the Pleistocene fluvial record. According to this last parameter and to the rarity of observed alluvial remnants, which prohibits precise longitudinal correlations, the studied area has been divided in three sectors according to the different local bedrock (Despriée *et al.*, 2004).

In the southern sector, located on the endogenous bedrock, several dam lakes mask the lowest part of the valley and only the higher alluvial outcrops have been observed. In a first time, four alluvial formations have been here recognized, respectively at + 90/ +110 m (sheet D), + 60/ + 75 m (sheet C), +45 / +50 m (sheet B) and + 15 / + 25 m (sheet A) of relative altitude above the floodplain. The ESR geochronological studies were realized on sediments carried out from the different terraces. Results show a good reproducibility and generally a good coherence with the altimetry, except for the highest formation where two groups of age were obtained. It has permitted to propose the existence of two different terraces, in spite of similar altitudes (Voinchet, 2002). New field works and sedimentological data have allowed to confirm this split. The similar altitudes of the two terraces are related to a recent faulting which has led to a 15 to 20 m down movement of the more ancient alluvial formation, called sheet E.

According to the ESR results (fig. 5), the beginning of the Creuse Valley incision and the first alluvial deposit

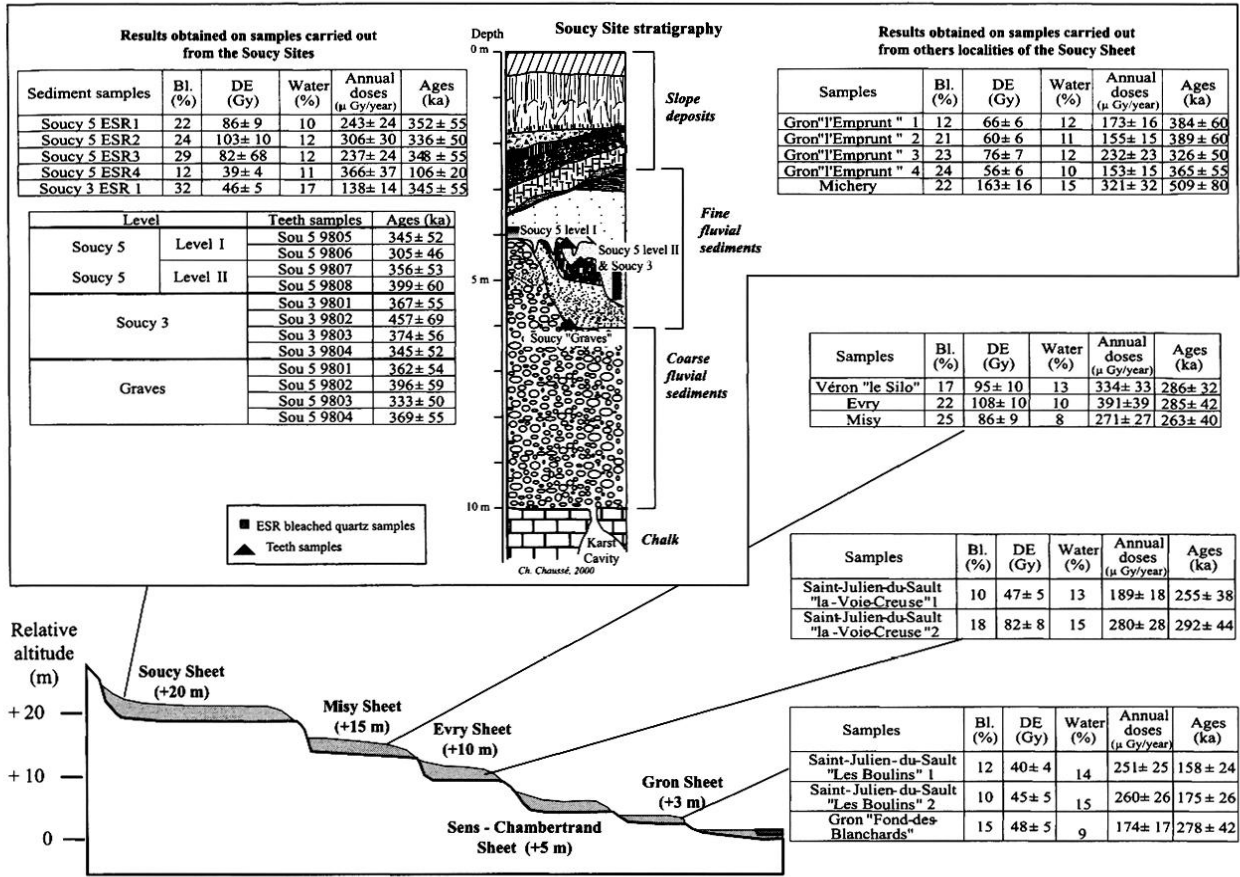


Fig. 4: ESR results obtained for sediments and teeth carried out from the lower terraces of the Yonne River system (France). The tables showing ESR results derived from quartz dating indicates for each analysed sample the following parameters: maximal bleaching percentage (Bl.), equivalent archaeological dose (DE), water content of the sediment, annual dose-rate and obtained age.
Fig. 4 : Résultats RPE obtenus pour des sédiments et des dents provenant des terrasses inférieures du système de l'Yonne (France). Les tableaux présentant les résultats RPE obtenus à partir de la datation de quartz, indiquent pour chaque échantillon analysé les paramètres suivants : pourcentage de blanchiment maximal (Bl.), dose archéologique équivalente (DE), teneur en eau du sédiment, dose annuelle et âge obtenu.

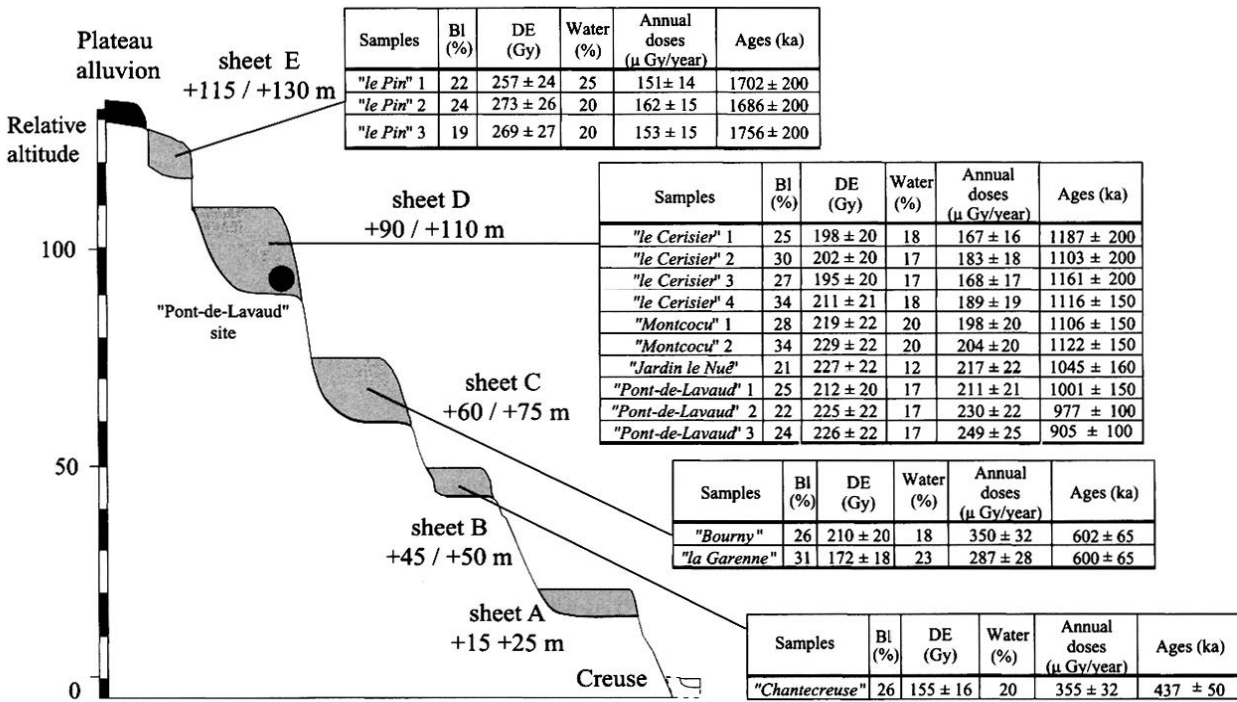


Fig. 5: ESR results obtained for sediments sampled in the "Massif Central" sector of the Creuse River system (France). The tables showing ESR results indicates for each analysed sample the following parameters: maximal bleaching percentage (Bl.), equivalent archaeological dose (DE), water content of the sediment, annual dose-rate and obtained age.
Fig. 5 : Résultats RPE obtenus pour des sédiments provenant du secteur " Massif Central " du système de la Creuse (France). Les tableaux présentant les résultats RPE indiquent pour chaque échantillon analysé les paramètres suivants : pourcentage de blanchiment maximal (Bl.), dose archéologique équivalente (DE), teneur en eau du sédiment, dose annuelle et âge obtenu.

are dated around 1.7 Ma B.P. Samples carried out from the Sheet D gave systematic Lower Pleistocene ages, ranging from 0.9 to 1.2 Ma and place the Early Palaeolithic site of "Pont-de-Lavaud" discovered in the alluvial formation amongst the most ancient archaeological localities of Europa (Despriée *et al.*, 2004).

CONCLUSION

The recent development of the ESR method applied to optically bleached quartz and its systematic use on alluvial deposits have allowed to elaborate chronostratigraphical frameworks on several Pleistocene fluvial systems of France.

The study of the behaviour of the ESR Al-center in quartz indicates that the alluvial sediments can be totally bleached in a recent deposit while an approximately six months exposure to solar light is necessary. It validates the hypothesis of a natural optimal bleaching and so the application of the ESR method to provide reliable ages on this type of material.

An ESR age calculation necessitates therefore not only the classical determination of both total and annual doses, but also the obtaining of the residual dose corresponding to the non-bleached part of the Al-signal intensity. It implies the study of the light-exposure response of each analysable sample and the precise determination of the maximal bleaching percentage.

In spite of this heavier analytical procedure, the use of the ESR method applied on alluvial quartz permit to furnish geochronological data on geological systems, which are difficultly dated by classical methods.

The ESR method was successfully applied on sediments carried out from the Yonne River and Creuse River systems. On the Middle Pleistocene Soucy site, the comparison between ESR results obtained on bleached quartz and ESR/U-series data derived from tooth enamel (around 350 ka) demonstrate the reliability of the ESR method applied on fluvial sediments and show hence the possibility to precisely date such archaeological and/or geological levels.

However, the complexity of the alluvial deposits constrains often to divide the studied fluvial systems in reduced geographical areas of similar characteristics, in order to minimize the effect of local tectonic events or various bedrock lithologies. The ESR analyses must be imperatively included in extensive studies of the considered fluvial systems including more particularly geological, sedimentological, palaeoenvironmental and magnetostratigraphical studies. A such study was realized on the Creuse River system, allowing the establishment of a synthetic framework for this valley, which has delivered an important early paleolithic site, dated by ESR of 1.1 Ma B.P. This study also focalised the great importance of the sampling. The accuracy and the reliability of the measurements depend of the sample choices and alluvial sheets, where significant hydric circulations or contaminations by slope deposits or periglacial phenomenon are observed, should be eliminated.

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