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ESR DOSE RESPONSE OF THE Al CENTER MEASURED IN QUARTZ SAMPLES FROM THE YELLOW RIVER (CHINA): IMPLICATIONS FOR THE DATING OF UPPER PLEISTOCENE SEDIMENT

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Abstract: The ESR dating method requires to describe the evolution of the ESR signal intensities *vs.* increasing gamma doses, then to extrapolate the equivalent dose of radiation received by the sample since its deposition using mathematical fitting. The function classically used to describe the growth curves of ESR aluminium signal in quartz was recently discussed and challenged for Lower Pleistocene sediments. In the present work, some alluvial sediments sampled in Upper Pleistocene fluvial terraces of the Yellow River system (China) permit us to test the application of another extrapolation function (linear + exponential) recently proposed for Lower Pleistocene sediments. The equivalent doses obtained here for the recent deposits of the Yellow River system and the corresponding ages are promising and indicate the potential of ESR to date quartz deposits from Upper Pleistocene times.

Keywords: ESR dating method, quartz, Upper Pleistocene, equivalent dose determination, exponential plus linear function.

1. INTRODUCTION

ESR is a classical dating method used in Quaternary Geology and Prehistory, particularly in the geochronological studies of alluvial terraces and associated prehistoric sites (Bahain *et al.*, 2002; Despriée *et al.*, 2011). One of the major interests of the method is to allow the dating of sediments deposited during the whole Lower and Middle Pleistocene. The ESR method is particularly suitable when seeking to date whole systems of alluvial terraces, set up following the climate changes and tectonic movements that have occurred throughout the Quaternary. The systematic dating of this type of stepped terraces system, using always the same method and analytical protocol, permits to easily compare the results between them and thus to reconstruct the evolution of the valley over a long period (Laurent *et al.,* 1998; Voinchet *et al.*, 2010, Tissoux *et al.*, 2011).

Until recently, the dating by ESR of the lowest terrace levels of a fluvial system was considered not to be working properly. Indeed, the ages calculated for the lower terraces of several French fluvial systems were systematically overestimated and were rarely younger than 200 ka, in disagreement with the other available geochronological data (see for example in Laurent *et al.*, 1998, Voinchet, 2002). For a long time, we explained that by the fact that these overestimated ages were due to the poor light sensitivity of aluminium (Al) center, used to determine the equivalent doses (D_E) and therefore ages.

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An experimental study of artificial optical bleaching on sedimentary quartz has showed that the Al centre was maximally bleached after a 6 months natural light equivalent illumination (Voinchet *et al.*, 2003). This value may seem too long to be observed in nature resulting in an incomplete bleaching, varying from one sample to the other. Then, we can suspect that a part of the measured signal could be related to this non-bleached dose (residual dose, D_r) that could explain the overestimation of the D_E and ages. This unbleached dose is relatively low for old samples in comparison with their high accumulated dose D_A ($D_E = D_A + D_r$, $D_A >> D_r$). However, for recent samples, the unbleached dose can be very important in comparison with the low accumulated dose ($D_E = D_A + D_r \approx D_r$, $D_r >> D_A$).

On the other hand, a study performed on present-day fluvial deposits has permitted to check that the maximal bleaching was reached after a transportation of grain for a few kilometers (Voinchet *et al.*, 2007). These grains are completely bleached and display only a residual signal associated with the presence of non-bleachable Al-centers (*deep aluminium traps*, DAT in Tissoux *et al.*, 2012).

Moreover, studies regarding the behavior of the Al-centers ESR signal in quartz in response to thermal treatment, optical bleaching or gamma irradiation (Toyoda and Ikeya, 1994; Voinchet *et al.*, 2003, Tissoux *et al.*, 2012) have demonstrated the existence of different components with different growth or disappearance kinetics. It implies a new way to determine the D_E before the age calculation. The single saturating exponential function initially used to describe the growth curves of Al center ESR intensities *vs.* increasing radiation doses (Yokoyama *et al.*, 1985) failed in the case of recent deposits. The use of this equation was recently discussed, mainly for Early and Middle Pleistocene samples (Duval *et al.*, 2011; Moreno, 2011; Cordier *et al.*, 2012; Duval, 2012), and the use of more complex function was proposed but never applied to date Upper Pleistocene deposits.

The present study focuses on the dating of late Middle Pleistocene and Upper Pleistocene terraces, which form the last and lower levels of Quaternary fluvial systems, using the example of Yellow River system, China.

The fluvial system of the Yellow River in Zhongwei area (Ningxia Province, China) (**Fig. 1**) seemed particularly suited to such research concerning the dating of "recent" fluvial terraces. Indeed, it is composed of a set of 11 terraces stretches since the current alluvial flood plain up to 130 m of relative altitude (Xing *et al.*, 2002). The lower terraces are clearly identified in the landscape and the entire region was little affected by human activity.

In order to date the Yellow River system by ESR, several sediments have been sampled in fluvial terraces attributed to Upper Pleistocene in November 2010, giving us the opportunity of such fitting studies. In this paper, the main results and conclusions of this study are presented.

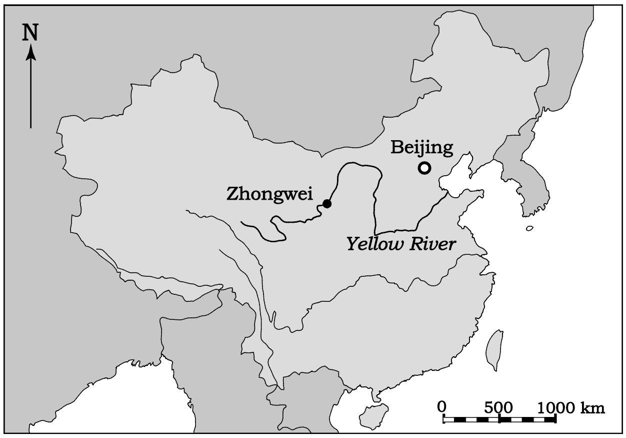

Fig. 1. *Location of the studied area.*

2. MATERIAL AND METHOD

Sampling

The Yellow river alluvial system, close to Zhongwei city, Ningxia Province, central China, is composed of a set of several stepped terraces, clearly observable in this semi-desert area. On the eleven levels described by Xing *et al.* (2002), only nine terraces were recognized during the present study (**Fig. 2**). It is therefore impossible to correlate easily the two described systems. Moreover, the studied area at present does not allow the access to the lowest alluvial formation described by Xing, due to the presence of a dam downstream. Additionally, our T3 seems to be a morphological terrace and only slope deposits were observed. This level was not sampled. Finally, we have not observed the highest terrace (T11) in the field.

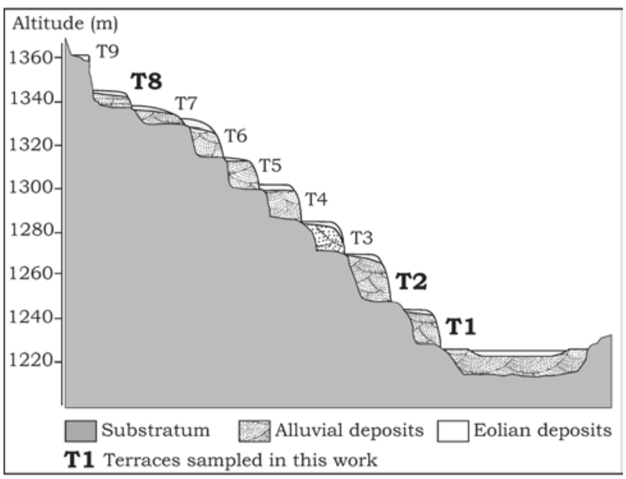

Fig. 2. *Terraces system of Yellow River in the Zhongwei area.*

The two lower terraces (T1 and T2) of our alluvial system are located a few meters above the current level of the river and their installation is estimated to date from the late Pleistocene. T1 is located between +1/+10 m above the current river bed (relative altimetry). T2 is +12/+17 m of relative height. T1 deposits consist of fine sands, sometimes silty. T2 sediments are fine sands.

Despite these supposed young ages, we have sampled these two alluvial formations at the same time then the other older fluvial remnants, in order to obtain a global view of the valley evolution. Three samples were taken in the terrace T1 (YR 05, YR 07 and YR 18), and four in the terrace T2 (YR 02, YR 03, YR 04 and YR 20).

A sample was also taken in one of the highest observed terraces of the system (T8, sample YR 16) in order to compare its dose response with the growth curves of the recent sediments.

ESR dating method of bleached fluvial quartz using the Al center

ESR is a paleodosimetric dating method. For this kind of method, samples are considered as dosimeters, which have recorded the total dose of ionising radiation received during the burial. This value can be estimated by the "additive dose method". The sample is separated into aliquots which are irradiated with increasing gamma doses. The additive method requires building a curve describing the evolution of the ESR signal as a function of increasing gamma doses, and then to extrapolate it to its intersection point with the x-axis. Then, the total dose is equivalent to the value directly read on the x-axis.

Analytical protocol and age calculation

Quartz is separated using a chemical and physical protocol already detailed in literature (e.g., Yokoyama *et al.*, 1985; Voinchet *et al.,* 2004). Irradiation of the aliquots is performed using a panoramic ^{60}Co source (Dolo *et al.*, 1996) emitting 1.25 MeV γ-rays with a dose rate of 200 Gy/h. Nine aliquots were submitted to gamma doses ranging between 260 and 12,000 Gy.

ESR analyses were done on 100-200 µm quartz grains and we have chosen to use the Al paramagnetic center. This centre is optically bleached during the fluvial transport. The bleaching is not complete and the residual unbleachable signal must be determined and then subtracted from the ESR response prior to any age calculation. The determination of the proportion of non-bleachable deep centres (DAT) was performed using a solar simulator SOL2. The light intensity then received by each aliquot ranges between 3.2 and 3.4x10^5 lux and samples were illuminated for 1600 h. The bleached aliquots ESR intensity was subtracted from the intensities of other aliquots (including natural) before the construction of the growth curve.

ESR measurements were performed at 107 K with a Bruker EMX spectrometer using the experimental conditions proposed by Voinchet *et al.* (2004). The signal intensity is measured between the top of the first peak at g=2.018 and the bottom of the 16th peak at g=2.002 of the Al hyperfine structure (Toyoda and Falguères, 2003). Each aliquot was measured three times after a rotation of an angle of 120° from its initial position in the cavity. This protocol was repeated three times representing 9 measurements for each aliquot.

The regression curves were obtained using the Microcal OriginPro 8 software. Data were weighted by the inverse of the squared intensity, $1/I^2$ (Yokoyama *et al.* 1985).

The dose rate was calculated from the radionuclide activities obtained by gamma-ray spectrometry measurements in situ (Inspector 1000, Canberra) using the threshold method of Mercier and Falguères (2007) and in our laboratory (Ortec low background germanium spectrometer). Age calculations were assessed using the dose-rate conversions factors from Adamiec and Aitken (1998), a k-value of 0.15±0.1 (Yokoyama *et al.*, 1985; Laurent *et al.*, 1998), α and β attenuation factors from Brennan (2003) and Brennan *et al.* (1991), water attenuation formulae from Grün (1994). The water content is measured by the difference in mass between the natural sample and the same sample dried for a week.

The cosmic dose rate contribution was calculated from the equations of Prescott and Hutton (1994). The internal dose rate was considered as negligible because of the low contents of radionuclides usually found in quartz grains (Murray and Roberts, 1997; Vandenberghe *et al.*, 2008).

Dose response of Al-center in quartz

Since the first optically bleached quartz grains dating by ESR (Yokoyama *et al.*, 1985), a single saturating exponential function (SSE) (Ikeya, 1981; Apers *et al.*, 1981) has been systematically used to fit the Al center dose response curve:

$$I(D) = I_{sat}(1 - e^{-\mu(D+D_E)}) \quad (1)$$

where I is the intensity of the ESR signal of a sample irradiated at the dose D, I_{sat} the saturation intensity, μ the coefficient of sensitivity of the sample and D_E the equivalent dose.

In this mathematical approach, while the number of trapped electrons increases, the possibility of a new electron trapping decreases and the curve approach asymptotically to a maximum intensity value (I_{sat}) that corresponds to the saturation of the system.

The analysis of quartz samples from terraces of very different ages shows however that the SSE formula, does not fully describe the evolution of the ESR intensity during gamma irradiation of quartz grains (Duval, 2009).

For samples of ancient terraces (**Fig. 3**), this equation does not take into account the slope failures frequently observed in the growth curve, which increases the uncertainty in the determination of the D_E.

For recent samples, the curve constructed from the equation through a few points and does not fit at high doses, causing systematic overestimation of the D_E and a significant error (**Fig. 4**), as it had already been noted for Middle Pleistocene samples by Duval (2012). This phenomenon has been described for other dated materials such as tooth enamel (Duval *et al.*, 2009), and the use of another function corresponding to the sum of a SSE function and a linear term (E+L) was proposed for teeth then for quartz (Duval *et al.*, 2011; Moreno, 2011; Cordier *et al.*, 2012 and Duval, 2012), considering that the global ESR signal is the result of the presence of two components in the Al signal:

$$I(D) = I_{sat}(1 - e^{-\mu(D+D_E)}) + B(D + D_E) \quad (2)$$

where I is the intensity of the ESR signal of a sample irradiated at the dose D, I_{sat} the saturation intensity, μ the coefficient of sensitivity of the sample and D_E the equivalent dose. The linear term represents the filling of traps existing prior to artificial irradiation. It depends thus on this radiation dose and the equivalent dose and saturates far beyond the maximal added artificial dose. B represents the radiation sensitivity of this term.

Ages calculated using this new equation have been recently published for quartz of Lower Pleistocene and Middle Pleistocene ages (Duval *et al.* 2011; Moreno, 2011; Cordier *et al.*, 2012; Duval, 2012). The use of this equation has brought better accuracy for these ages and their use to date more recent sediments seems possible. In order to check this point, several sediment samples from Yellow River system were analyzed in the present work.

3. RESULTS AND DISCUSSIONS

U, Th, K concentrations, water content, estimated cosmic dose and bleaching rate are displayed in **Table 1**.

Equivalent doses (and ages) were determined using both fitting functions. Results are displayed in **Table 2**.

As mentioned previously for others samples, we observe very different kinetics of intensity growth for the ESR Al signal curve between recent samples (whose age is between 10 and 200 ka) and ancient samples for YR sediments.

- For very recent samples, two distinct domains of growth are clearly visible on the curves, with an initial extremely rapid growth for the first doses and later a much slower one for high doses. In such conditions, using the classical SSE function to determine the equivalent dose systematically leads to an overestimation of the equivalent dose and, consequently, the age (**Fig. 4**). This scheme in two domains, with a very rapid growth and then linearity, shows before any calculation that the sediment would provide relatively

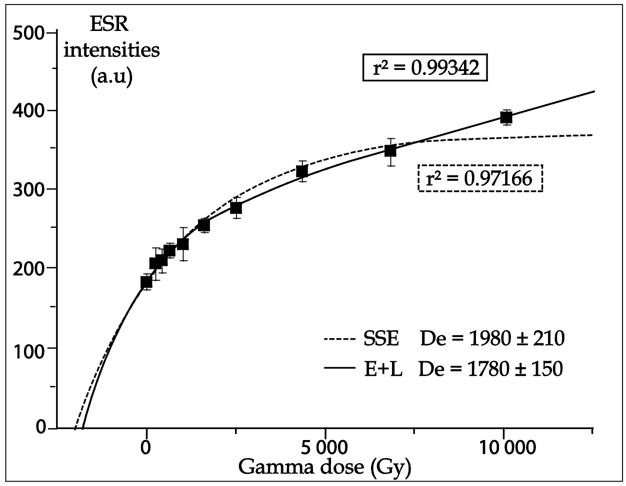

Fig. 3. *Growth curve of YR 16 sample, comparison of the two different fitting equations. Errors in every points correspond to the nine measures standard deviation.*

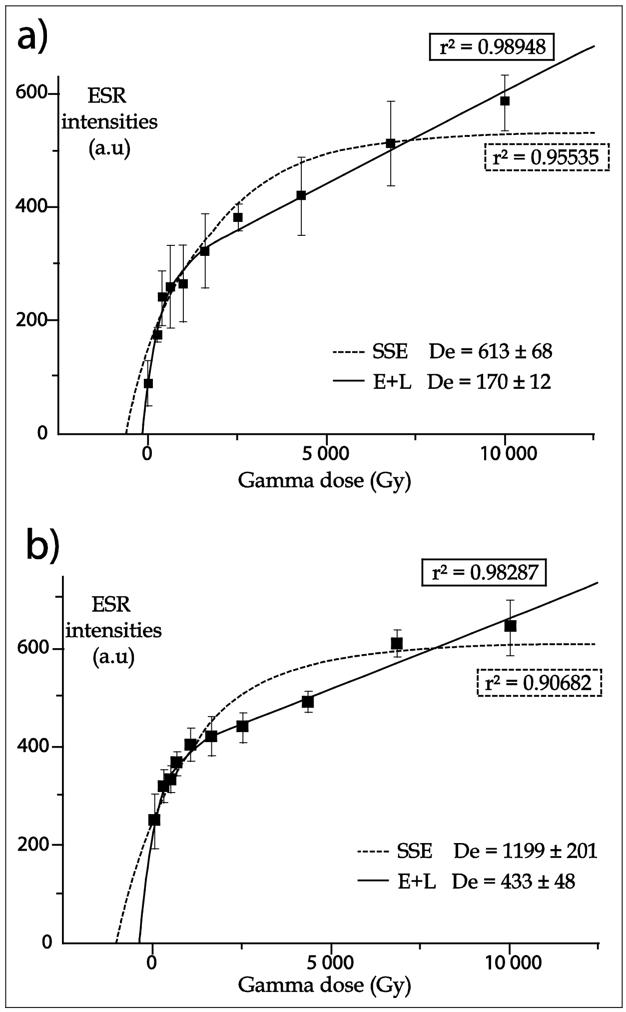

Fig. 4. *a) Growth curve of YR 07 sample, comparison of the two different fitting equations; b) Growth curve of YR 03 sample, comparison of the two different fitting equations. Errors in every points correspond to the nine measures standard deviation.*

Table 1. *Radiometric data obtained for the samples taken in the alluvial terraces of Yellow river system. The following analytical data are showed: U, Ra, Rn, Th and K activities, maximal bleaching percentage (Bl.), sediment water content (W) and estimated cosmic dose.*

Terrace	Sample	U (dpm/g)	Ra (dpm/g)	Rn (dpm/g)	Th (dpm/g)	K (%)	Bl (%)	W (%)	Cosmic Dose (µGy/an)
T1	YR 05	1.333±0.07	1.291±0.148	1.162±0.022	1.547±0.035	1.744±0.019	49	18	167±8
	YR 07	1.771±0.07	1.732±0.127	1.547±0.021	1.877±0.032	1.872±0.056	41	17	181±9
	YR 18	2.036±0.07	1.655±0.127	1.766±0.022	2.239±0.032	2.045±0.056	39	17	197±10
T2	YR 02	1.675±0.06	2.002±0.120	1.805±0.021	1.792±0.029	1.517±0.014	47	18	200±10
	YR 03	2.059±0.06	1.759±0.115	1.935±0.020	1.960±0.028	1.438±0.013	46	20	105±5
	YR 04	2.565±0.08	2.289±0.160	2.071±0.028	1.974±0.039	1.455±0.018	53	20	181±9
	YR 20	1.942±0.05	1.657±0.110	1.648±0.018	2.119±0.028	1.692±0.013	53	12	62±3
T8	YR 16	1.771±0.04	1.648±0.087	1.531±0.014	1.802±0.021	1.315±0.009	53	17	167±8

Table 2. *ESR data and ages obtained for the samples taken in the alluvial terraces of Yellow river system. The following analytical data are showed: equivalent dose obtained using SSE fitting (D_E SSE) and using E+L fitting (D_E E+L), dose rate and obtained age with the two fitting procedure.*

Terrace	Sample	Dose rate (µGy/an)	De SSE (Gy)	Ages SSE (ka)	De E+L (Gy)	Ages E+L (ka)
T1	YR 05	2805±279	993±53	354±40	225±14	80±9
	YR 07	2998±280	613±68	203±23	170±12	57±6
	YR 18	3495±302	736±71	210±26	185±15	53±5
T2	YR 02	2568±241	1188±152	463±50	498±60	194±29
	YR 03	2608±260	1199±201	460±60	433±47	166±25
	YR 04	2957±280	2761±141	933±103	2395±130	810±98
	YR 20	2816±270	1272±260	451±52	506±80	180±27
T8	YR 16	2311±230	1980±212	856±86	1645±90	712±75

recent age (late Middle Pleistocene or Upper Pleistocene).

- For old sample, the curve does not clearly show different growth domains, and applying the SSE equation or E+L equation is statistically indistinguishable (**Fig. 3**). However, the use of E+L gives a smaller error range and generally a smaller D_E value (3-5%).

We can note that the results obtained using the two methods for the fitting show some similarities, essentially in terms of homogeneity within the different terraces. Two age groups are then obtained for the T1. Ages provided by YR 07 and YR 18 samples are very close and YR05 seems older. This age seems to be overestimated, especially for the terrace situated a few meters above the current level of the river. The three samples have the same relative altitude and there is no evidence of fault movement or slipping to explain simply D_E 5 to 6 times greater than those observed for the other remnants to the terrace.

This age could indicate that the sediment had not been sufficiently bleached prior to its deposition or a contamination of the sediment with unbleached grains. Indeed, when a quartz grain is deposited without being completely bleached, the residual dose will add up to the accumulated dose during the burial and thus increase the value of the calculated age. In this case, for recent sediment incompletely bleached prior to deposition, the accumulated dose (since the deposition) is small in comparison to the residual dose. The induced error or overestimation in D_E determination is then very significant and would explain overestimation of the age obtained here. The risks of insufficient bleaching seem important for silty or silty-sandy deposits. The transportation of grains in turbid water could in fact prevent the penetration of UV in deep water and limit their action on sands.

For the T2 terrace, two groups are also identified. YR 02, YR 03 and YR 20 show consistent age (around 180 ka), whereas YR 04 was significantly older, and its age was probably overestimated. The growth curve of this sample is very similar to those obtained for the samples from the highest terraces (no rapid growth for the lowest doses and so high D_E). Sedimentological analysis revealed in the corresponding sediment the presence of aeolian quartz grains (quartz grains morphoscopy). Unlike the other samples where the selected grain size quartz has single origin, the grains of this sample probably correspond to two different deposition ways. The two subsequent populations of grains do not have the same story and not the same characteristic. The presence of these grains in the sample makes impossible the building of a significant growth curve. It can explain the overestimated age of the sample YR 04.

Nevertheless, there are important differences between the two sets of results obtained using the two ways of D_E calculation. These results result in very different conclusions for the reconstruction of the history of the alluvial system. Hence in the present case, if we consider the SSE ages, the Yellow River stepped system history will be mainly of Middle Pleistocene age and strongly affected by the tectonic uplift, without evolution during the last 200 ka, in disagreement with other stratigraphical and palaeontological evidences (see for exemple Porter *et al.*, 1992; Yin *et al.*, 2013). If we consider the second case (E+L ages), the incision has continued during the whole Middle Pleistocene and the Upper Pleistocene until very recently. This evolution indicates a continuous regional uplift since the early Middle Pleistocene and place the onset of the stepped system close to the Lower-Middle Pleistocene limit, ca. 780 ka ago.

Comparisons between our results and ages obtained through other methods (ESR measurement using Ti center, OSL) that are being implemented will help to better define the context of the implementation of the Yellow River system.

4. CONCLUSION

The geochronological results obtained for the Quaternary alluvial deposits of the Yellow river illustrate clearly that during irradiation of samples of quartz, there are several answers of the Al center depending on the added doses. This phenomenon is much more noticeable for samples with small D_E, and hence for very recent samples (late Middle Pleistocene and Upper Pleistocene).

The use of several terms regression functions allows us to take this phenomenon into account, and provides dates for deposits of less than 100 ka. Ages obtained for lowest levels indicate the potential of ESR in quartz to date samples from the Upper Pleistocene. These results open new perspectives for samples until now considered non-datable.

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