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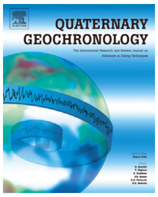

Evaluation of ESR residual dose in quartz modern samples, an investigation on environmental dependence

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ABSTRACT

Luminescence and ESR dating methods of quartz sediment are based on the natural resetting of the signal by light exposure (optical bleaching). When the bleaching is incomplete, a residual dose (D_{eR}) is added to the post-depositional dose accumulated since the deposit and hence the age is overestimated.

Insufficient bleaching is usually linked to the environment and conditions of transport/deposition of the quartz grains affecting the light exposure duration. Indeed, each transportation mode – fluvial, marine or aeolian – is associated to specific conditions of light exposure, depending mainly to the location of grains in the transport agent during the transport phase, the opacity of the transport environment and the velocity of the transport.

The present study attempts to discriminate the modes of transport/deposition providing a satisfying reset of the ESR signals of quartz grains. For this purpose, we investigated bleaching rates and ESR residual doses of aluminum centers from "present-day" aeolian, fluvial and marine sediments sampled in various sedimentary environments. The bleaching efficiency evaluation in these different environments may help for a better understanding of the resetting phenomenon for quartz signals which represents presently the main difficulty for ESR dating.

The results show that the residual doses are small enough to allow an ESR dating of the main part of the sediment transported in almost all the context examined in this study. The smallest residual doses are obtained from quartz grains within the range of 100–200 μm and transported in clear water. Some limits for the application of optically bleached quartz ESR dating appears nevertheless, mainly when the residual dose and the dose accumulated after the deposit are quite similar, i.e. for Upper Pleistocene samples.

1. Introduction

Quartz is a very common mineral component of Quaternary continental deposits such as fluvial, lacustrine, marine or aeolian sediments. These deposits have often recorded climate and cultural changes of the last few million years and represent therefore major archaeological research areas because the rivers banks, lakes or marine beaches have always been preferred areas of human bearing occupations. Hence, their dating presents an interest to geologists and geographers, and also for prehistorians and paleoanthropologists (Falguères, 2003; Voinchet et al., 2003; Duval, 2012).

ESR method is based on the following physical principle: natural radiations delocalize electrons within the quartz lattice and they are later trapped in defects in the crystal system. These trapped electrons are then detected by ESR, which allows the estimation of the radiation dose received by the sample since its formation or its deposit in the case of the sedimentary quartz (accumulated dose).

For a geochronological application for sediments, the quartz geochronometer should be reset to zero. For sediments, this reset called optical bleaching is made by the action of sunlight on the

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quartz during the phase of transport prior to the deposition. In case of fluvial transport, this resetting is practically obtained after few kilometres (Voinchet et al., 2007). Indeed if this bleaching is incomplete, a residual dose (D_{eR}) added to the accumulated dose since the deposit, known as post-deposit dose or archaeological dose, will contribute to overestimate the age value (Fig. 1). Younger is a deposit, higher will be the impact of the residual dose on the age (overestimation).

The use of aluminum center for ESR dating increases this problem. This center is not much sensitive, both to bleaching effect (unbleachable part) and to irradiation (the dose response of Al signal increases slowly and doesn't show any saturation in natural condition). This low sensitivity is useful for dating old deposits because the aluminum signals almost never saturate, but it induced however a real risk of poor bleaching during the transport phase of quartz grains.

This insufficient bleaching is usually linked to the conditions of transport and deposition of the quartz grains. There is for example a possibility of incomplete bleaching if the time of light exposure is too short or if the light intensity is too low. These bleaching conditions are directly dependent of the modes of transportation.

In the case of water transportation, clear water, cloudy water rich in microorganisms or low-suspended sediment, or muddy water, very rich in silt and clays, will influence directly the bleaching quality. Each of these environments will create conditions more or less conducive to the light exposure of the grain in suspension or traveling on the bottom.

In the case of a transport by wind, light intensity passing through the cloud of particles in suspension in the air will depend on their size and the size of the cloud of sediment. The bleaching of sediment will be different for a sand wind, creating dunes, or in fine particles clouds as in the case of loess.

Could ESR date every kind of sediment? Which transportation conditions are the most conducive to obtain a maximum bleaching? Does the grain size have an effect on the bleaching rate for the same transportation type? What is the impact of an incomplete bleaching on the age estimates? If the unbleached part is important, what is the induced age overestimation?

In order to solve these issues and to establish a protocol for selection of fossil sediments excluding the badly bleached deposits, we present here an evaluation of the residual dose of present-day samples, according to various depositional environments.

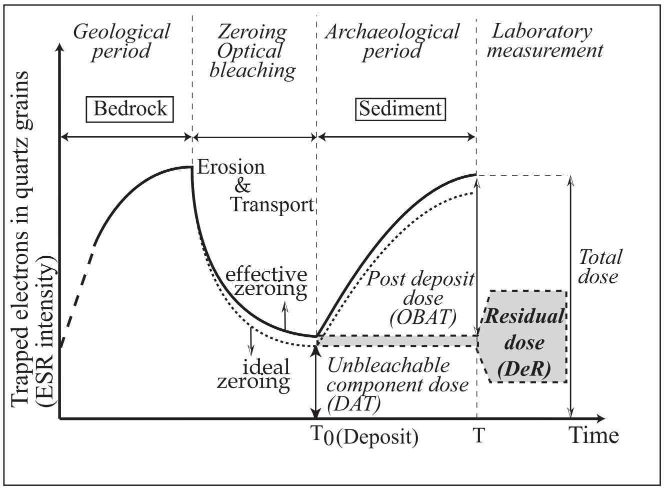

Fig. 1. Evolution of the dose recorded by the Aluminum centers during history of the quartz grain, identification of non-bleachable centers (DAT), post-depositional dose (OBAT), total dose and residual dose (D_{eR}); Highlight of the D_{eR} role in the overestimation of post-depositional dose and age.

In order to discriminate the modes of transport/deposition allowing a good reset of ESR quartz signals, bleaching rate of modern sediments was studied. In that aim, each sample was separated into two parts: one was exposed to artificial bleaching using a luminous device reproducing the solar spectrum and the other will be kept intact. The comparison of the ESR intensities of the two subsamples highlight the existence or the absence of a residual dose.

If a residual dose has been revealed, a determination of the equivalent dose and the apparent age calculation was made.

2. Sample selections

The samples selected for this study come from different sedimentary environments:

- Clear water fluvial sediments of lowland river from temperate zone,
- Silty water fluvial sediments from estuary area,
- Marine beach sediments,
- Shoreline dunes,
- Aeolian sands extracted from desert dunes.

2.1. Grain size dependence

The grain size has an implication in the way of transportation (Fig. 2). The finer sandy sediments have a fast transport in suspension, usually in mass, localized in the top of the transport agent (water or air). In medium or large size rivers, these sediments are transported only once and are deposited virtually only at the level of the lower river course and estuary. The coarser sediments are transported more slowly, usually in several phases with a new displacement at each river efficiency increase, mainly linked to episodes of high water or overflow occurring seasonally each year in the region of high and middle latitudes. The coarser grains are transported in the lower part of the transport media (traction-

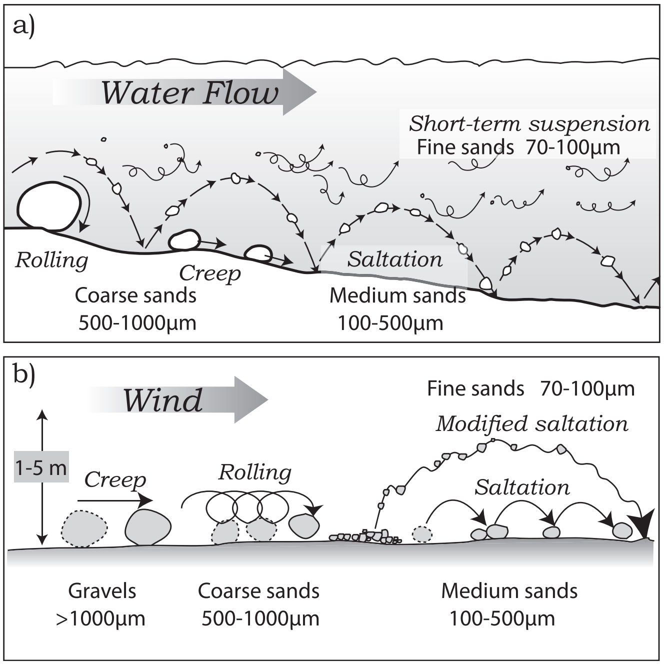

Fig. 2. Transport modes of different grain size sands by water (a) and by wind (b) (modified from Kok et al., 2012).

rolling-saltation). The intermediate particle size corresponding to the grains usually used for dating, have an intermediate behavior (saltation-suspension). Therefore, for each environment, different modes of transport exist, according with the different grain sizes, leading to different light exposure conditions, according to the position of grain in the carriage thickness and taking into account single or multiple transports.

In order to determine the grain size dependence, three different grain size fractions were selected when it was possible (60–100 μm, 100–200 μm and 200–500 μm). For most of our samples, finest grains were lacking and only the two coarser grain size fractions (100–200 μm and 200–500 μm) could be extracted.

2.2. *Fluvial sediments*

In order to evaluate the influence of the opacity of the river water on the zeroing of paramagnetic centers, we have sampled sediments from translucent clear water and opaque silty water. The study of these samples should demonstrate if changes in the river efficiency can induce difference in the sediments bleaching.

The sampling of present day sediment deposited in clear water river was performed in the middle course of the Loire River, near the city of Orléans (France). The sample comes from a beach unwatered by the decrease of the river during the summer.

Regarding the turbid water river, the samples were collected in the lower course of the Garonne River near the city of La Réole (France). Several samples have been taken in sediment deposited at various time of a same flood episode: during the primary flood (overflow sample), and at the end of this event (overflow end sample). Sediments reworked by the river were also collected.

2.3. *Marine sediments*

Marine sediments come from Noirmoutier Island, located on the Atlantic French coast, in an area where there is significant recent movements of the beaches. The southwestern edge of the island is formed by sands deposits due to accretion or regression based on tides or storms.

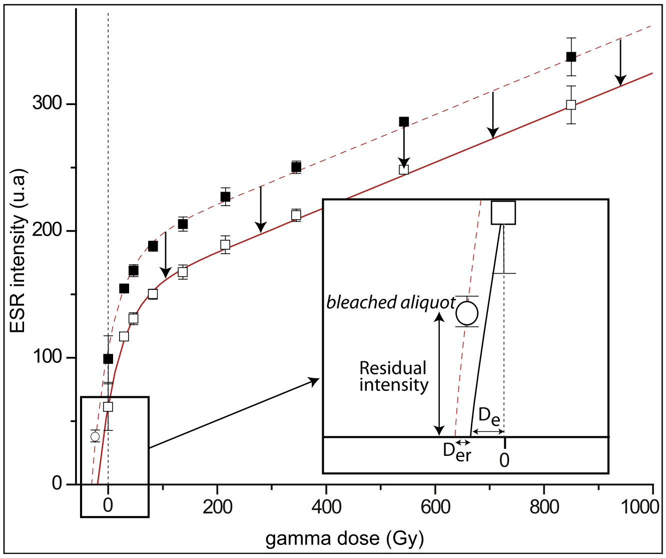

Fig. 3. Dose response curve and graphical representation of De evaluation with I_{bl} substract. White circle represents the ESR intensity of bleached aliquot; black squares represents ESR intensity of natural and irradiated aliquots without I_{bl} subtracting; white squares represents ESR intensity of natural and irradiated aliquots after I_{bl} subtracting. Continue curve represent the Dose response curve after I_{bl} subtraction.

We selected two sediments. The first sample has been taken directly in current beach sands, moved by each tide. A second sample was collected in a dune located close to the shoreline. This dune, located 800 m inland, could correspond to an older sea level or to the reworking process induced by the wind. The sample was collected at around five centimeters bellow the actual topography and a grass roots stratigraphic level.

For these two sediments, only two grain sizes have been studied: 100–200 μm – 200–500 μm. The 60–100 μm grain size fraction was not enough abundant to be investigated.

2.4. *Aeolian sands*

Aeolian samples have been extracted from Merzouga dunes in the southeast part of Morocco close to the Algerian border. An *erg* of about 30 km wide is constituted at this place by a number of sand dunes.

Two different samples of sediments were taken: the first one at the top of modern dune (upper layer); the second one after digging on about one meter inside the dune (inner layer).

One more time, only the two coarser grain sizes have been extracted. The 60–100 μm fraction was not abundant enough to be investigated.

3. Analytical protocols

3.1. *Quartz extraction*

Quartz was separated using a chemical and physical protocol already detailed in the literature (Yokoyama et al., 1985; Voinchet et al., 2004). Irradiation of the aliquots was performed using a panoramic ^{60}Co source (Dolo et al., 1996) emitting a 1.25 MeV -ray with a dose rate of 200 Gy/h. Nine aliquots were submitted to gamma doses ranging between 30 and 900 Gy.

3.2. *Bleaching and bleaching rate determination*

ESR studies were performed using the Aluminum (Al) paramagnetic center. This centre cannot be completely optically bleached during the fluvial transport and residual unbleachable signal intensity must be determined for each sample and then subtracted to the ESR signal response prior to any age calculation. The determination of the proportion of non-bleachable Deep Aluminum Traps (DAT) (Tissoux et al., 2012) is realized with a solar simulator SOL2. The light intensity received by each aliquot ranges between 3.2 and $3.4 \cdot 10^5$ Lux and samples are illuminated during 1600 h. The bleached aliquots ESR intensity is subtracted from the intensities of other aliquots (including natural) before the construction of the growth curve (Fig. 3).

The determination of the bleaching rate (δ_{Bl} expressed in %) is performed by comparing the natural ESR intensity (I_{nat}) and ESR intensity after artificial bleaching (I_{bl}), using the following formula.

$$\delta_{Bl} = ((I_{nat} - I_{bl})/I_{nat}) \times 100$$

A bleaching rate of 8% will mean that 8% of the total signal measured was bleachable by UV; the non-bleachable dose (DAT) represents then 92% of the measured dose.

3.3. *ESR measurements and equivalent dose determination*

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).

The D_E determination is based on the use of sum of an exponential function and a linear term. This function was proposed first for the teeth then for quartz (Duval et al., 2011; Moreno, 2011; Cordier et al., 2012; Duval, 2012) considering that the global ESR signal is the result of the presence of two components in the Al signal:

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

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 regression curves were obtained using Microcal OriginPro 8 software weighting the data according to the inverse of the intensity, $1/I^2$ (Yokoyama et al., 1985).

3.4. Apparent age calculation

Working on present-day deposits, having no actual thickness and not included in a succession of stratigraphic layers, poses a problem for the determination of apparent age. Indeed it is not possible to determine an annual dose comparable to that of fossilized sediments.

We have therefore chosen to use 'theoretical' annual doses determined in fossil deposits emplaced in comparable conditions to those studied (same transport mode and same geological context).

The dose rate of these comparable sediments (called D_a in the results) was calculated from the radionuclides activities obtained by gamma-ray spectrometry measurements *in situ* (Inspector 1000, Canberra) by threshold method (Mercier and Falguères, 2007) and in our laboratory (Ortec high resolution low background germanium detector). Alpha and beta attenuations were estimated from the tables respectively of Adamiec and Aitken (1998) and Brennan (2003). A k-value of 0.15 was used (Yokoyama et al., 1985; Laurent et al., 1998).

The cosmic dose rate was calculated from the equations of Prescott and Hutton (1994).

Dose rate (D_a) used for apparent age calculation are indicated in Table 1. Due to beta attenuation, in relation with grain size, specific D_a is given for each analyzed sub-sample.

Table 1
Bleaching rate (δ_{Bl} %), Residual dose (D_{eR}), theoretical dose rate (D_a) and apparent ages (+Ages) determined for the different grain size fractions of studied present-day fluvial, aeolian and marine sediments.

Samples		Grain size (μm)	δ_{Bl} (%)	D_{eR} (Gy)	D_a (μGy/a)	+Age (a)
Loire River		60–100	8	7	2600	2700
		100–200	4	4	2500	1600
		200–500	7	6	2430	2500
Garonne River	Overflow	60–100	13	19	3610	5270
		100–200	9	11	3500	3140
		200–500	11	12	3400	3540
	Overflow end	60–100	5	21	3610	5820
		100–200	5	5	3500	1430
		200–500	19	9	3400	2650
	Reworked sands	60–100	18	18	3610	4990
		100–200	22	28	3500	8000
		200–500	20	25	3400	7370
Morocco Dune	Upper sands	100–200	4	7	2000	3500
		200–500	14	16	1880	8490
	Inner sands	100–200	8	13	2000	6500
		200–500	12	18	1880	9550
Noirmoutier Island	Beach	100–200	4	4	2000	2000
		200–500	6	7	1880	3720
	Dune	100–200	10	15	2000	7500
		200–500	11	17	1880	9020

4. Results and discussion

The results concerning the different environments and the different investigated grain sizes are presented in Table 1 and Fig. 4 and show the bleaching rate (δ_{Bl}), the residual equivalent dose (D_{eR}), the estimated Dose rate (D_a) value and the apparent age (+Age) resulting of these doses.

4.1. Bleaching rate

Fluvial sediments: The δ_{Bl} determined for all the fluvial sediments shows the same trend: they never reach zero value and similarly vary with the grains size (Fig. 4a).

The lowest δ_{Bl} is observed for the medium grain size and the highest corresponding to the finest grains, except for the overflow end sample. δ_{Bl} determined for clear water range between 4 and 8% and are generally lower than those determined for the opaque water. For the silty water, δ_{Bl} range between 5 and 21%.

Reworked sands: The have completely different δ_{Bl} characteristics (medium grain size is higher than the two others fraction) and are not comparable to fluvial deposits *stricto sensu*.

Aeolian sediments: Only two grains sizes have been investigated for the Moroccan dune sands. The data indicate the same trend for the upper and inner sands, with the lowest δ_{Bl} obtained for the 100–200 μm fraction and the highest for the 200–500 μm fraction (Fig. 4b). However a difference exists, with much greater contrast in δ_{Bl} to the upper sand (4–18%) than in δ_{Bl} of the inner sand (8–12%).

Marine sediments: As for the aeolian deposit, the investigated marine sediments show similar trends, with the lowest δ_{Bl} obtained for the 100–200 μm fraction and the highest for the 200–500 μm fraction (Fig. 4b). However, whatever the grain size, there is a great difference between the bleaching rates of the two samples, the δ_{Bl} of the dune sands is two times higher than those of beach sands.

4.2. Residual dose (D_{eR})

The global trend of the entire D_{eR} follows that of the δ_{Bl}. Indeed, the D_{eR} is never equal to zero and, except for the reworked Garonne sediments, the lowest D_{eR} is observed for the medium grain size.

Fluvial sediments: The D_{eR} are systematically three to four times larger for the silty sediments than for the clear water samples (Fig. 4c).

The largest values are observed for the reworked sands. Alluvium *stricto sensu* present the lowest doses for sediments deposited at the end of the flood event and the highest dose for the sediment deposited during the flood.

The results obtained on the grain size dependence test in the clear water and in the silty water are comparable: the lowest D_{eR} is observed for the medium grain size, the highest corresponding to the finest grains.

Surprisingly, the quartz reworked by the flood event present the best bleached grains for the grain size fraction between 60 and 100 μm and the poorest bleached ones to the 100–200 μm fraction. This difference in comparison with the “alluvium *stricto sensu*” may indicate that the fine grains was transported by the river on a long journey while larger grains come from the erosion of older deposits, relatively close to the sampling place.

Marine sediments: For the two samples, as it was observed for the fluvial sediments, the 100–200 μm fraction yielded the

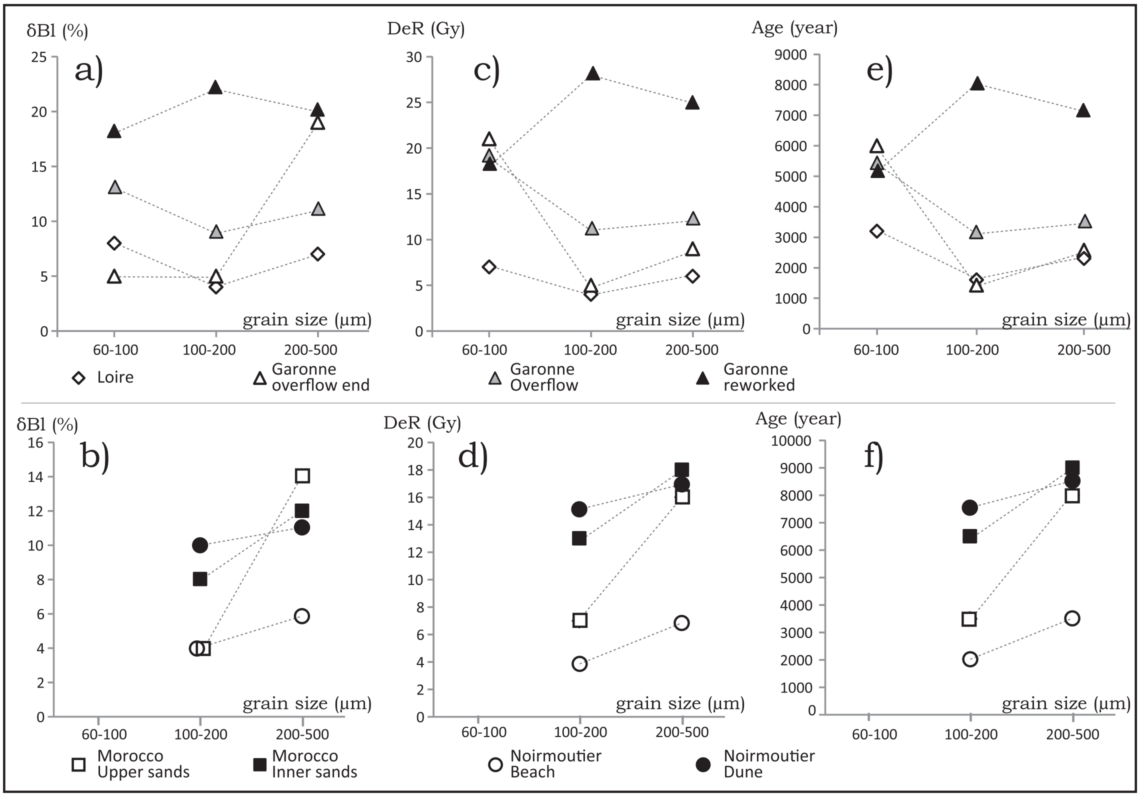

Fig. 4. Bleaching rate (δ_{Bl} %) (a, b), Residual dose (D_{eR}) (c, d) and apparent ages (+Ages) (e, f) determined for the different grain size fractions of fluvial, aeolian and marine sediments.

lowest D_{eR} (Fig. 4d). There is however an important difference in D_{eR} for samples extracted from the beach with those extracted from the dune, the D_{eR} of dune sands being three times higher.

Aeolian sediments: For the two aeolian samples, as for what it is observed for the fluvial and marine sediments, the D_{eR} determined for the 100–200 µm fraction is significantly lower than the D_{eR} of the 200–500 µm fraction (Fig. 4d).

We note here a great difference in D_{eR} for samples extracted from the upper sands or top-sands with those extracted from inner level of the dune. The D_{eR} of inner sands is two times higher.

4.3. *Apparent age*

In order to calculate apparent ages, annual doses measured in fossil sediments deposited in the same conditions and in the same region (same geological material) have been used (see section 3.2). The error range for the apparent ages (+Age in Table 1) is estimated to be 15%.

For fluvial clear water sediments, the apparent ages determined ranges between 1600 and 3200 years. These values correspond also to the smallest results obtained for turbid water ones (100–200 µm fraction of sands deposited at the end of the flood event). For overflow and reworked sands the apparent ages range between 3140 and 5430 years and between 5140 and 7140 years respectively (Fig. 4e).

Moroccan dune sands provide apparent ages around 3500 years for the upper layer and around 6500 years for inner layer (Fig. 4f).

As for the aeolian sediments, the apparent ages calculated for the two marine samples are clearly different. The beach yields an age around 2000 years and the dune age estimate is around 7500 years (Fig. 4f).

5. Discussion

5.1. *Grain size dependence*

Except for reworked fluvial sands, the lowest and highest D_{eR} and apparent ages are obtained for 100–200 µm grain size, and 60–100 µm grain size respectively.

This phenomenon can be explained by fine particles transport mode. These particles travel both in air and water in the form of a cloud. In this cloud, each grain is rarely, and for a short time, exposed directly to the light only when it travels at the top of the cloud. Hence the efficient light is quickly attenuated within the cloud thickness. Moreover, these fine particles, because of their low weight, are easy to carry and they travel more or less “nonstop”, in the poor conditions of light exposure as described here above, between their source place and their final deposit place.

In comparison, during the transfer from upstream to downstream, coarser grains are deposited as lateral sand bar were they are directly exposed to sun light. These sand bars are often displaced, mainly during flood. As for an example, Garonne sand bars move downstream of about 20–30 m by year (Malavoi and Bravard, 2010). In such conditions, coarser grains are exposed to light several times for long periods, before their final deposit in the sedimentary unit.

5.2. *Difference between clear and turbid fluvial waters*

Our data demonstrate that the bleaching of quartz grains is poor in opaque water where the UV cannot pass deeply through it. The difference observed between samples from overflow and overflow-end supports this observation. It is therefore possible to propose that the sedimentation mode, brutal and short event, is partly a

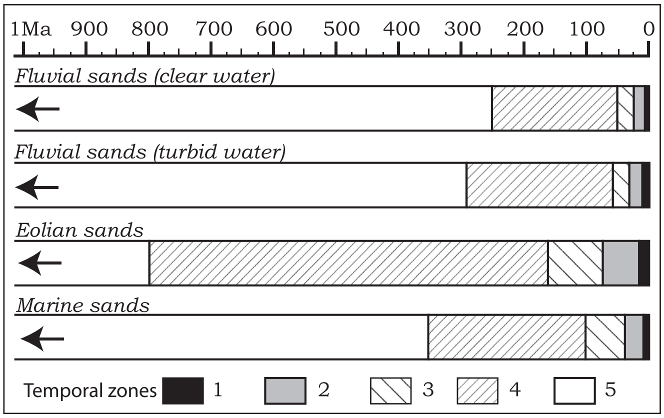

Fig. 5. Temporal zones defined by the relationship between the D_{er} and the total dose, for fluvial, aeolian and marine sediments. 1.) D_{eR} represents more than 25% of the total dose; 2.) D_{eR} values ranging between 10 and 25% of the total dose; 3.) D_{eR} represents between 5 and 10% of the total dose; 4.) D_{eR} represents between 1 and 5% of the total dose; 5.) D_{eR} represents less than 1% of the total dose.

cause of this result. Indeed, the sudden increase of river efficiency and velocity may lead to a decrease of the grain displacement time, both for fine and coarse grains, and therefore to a reduction of the corresponding light exposure times. This explanation supported by the fact that, when the efficiency decreases (overflow end), all the parameters determined for the Garonne turbid River become close to those of the Loire clear River.

5.3. *Marine sediments*

The difference in D_{eR} and apparent age observed for the two samples of Noimourtier Island seems greatly related to their sedimentation histories. The shoreline dune is really older than the beach, as revealed by the discovery of Iron Age sites within the dune (Ters et al., 1978). The apparent age of 7500–8500 years obtained for this sample seems therefore not only linked to a bad bleaching of the sediment, but also to its antiquity. If we correct this date by subtracting the 2000 years apparent age determined for the beach sediments, we get an age, about 5500–6500 B.P for the dune formation, yet slightly overestimated from the expected one.

5.4. *Aeolian sands*

The results obtained for the aeolian sands, and in particular the higher doses observed for the deepest levels have led us to recharacterize the studied dune. After discussion with the geologists who worked on this dune it appeared that the sampling has not been made in a dune in construction but rather in a dune being dismantled or in migration. This would explain why the upper sands are more exposed to the light and then better bleached than sands from the deeper layers, which have already recorded a post-depositional dose.

6. Conclusion

The results obtained during the study about the bleaching quality of present-day sands deposited in various sedimentary environments led us to define several recommendations concerning the choice of the most bleached sands for ESR dating.

The first point concerns the selection of the grain size fractions which undergone the best reset. Whatever the transportation mode, should be avoided too fine grain, mainly corresponding to quick single displacement in clouds, leading poor conditions of optical bleaching. The coarser grains (size bigger than 20 μm) moved by rolling and creep on the floor, are often less exposed to light during the phases of transport. Whatever the opacity of water, 100–200 μm fraction reveals therefore being most appropriate for ESR analyses.

The second point concerns the identification of transportation modes which provide best sediments bleaching. The sands carried by water seem to be better bleached at the time of their deposition than aeolian sands. Moreover, within the water transportation mode, the clearest transport environments should be preferred.

A residual dose (D_{eR}) has been evidenced in all the studied sediments. This dose is generally low in comparison to those observed in “fossil” sediments of the same depositional environments. For example, in Loire River, the youngest fossil terrace sampled close to our sampling location gave a D_e of 960Gy, which represents 192 times the residual dose observed in present day sediment. This low D_{er} nevertheless induces a small overestimation of the ages which must be taken into account.

This overestimation, corresponding to a few thousand years, led us to set upper limits on ESR dating application. Indeed, such overestimation will have a great impact if the real age of the deposit is young, for example for Upper Pleistocene sediments. On the contrary, for oldest sediments (middle or lower Pleistocene), this overestimation can be considered as minor or even negligible.

It is then possible to propose, for each transportation mode, different temporal zones in which the value of the residual dose, about few grays, allows or prevents the obtainment of a reliable ESR age (Fig. 5). If the D_{eR} represents more than 25% of the total dose (area 1 in black in Fig. 5), corresponding to deposits younger than 15–20 ka, ESR dating of optically bleached quartz using Al centers is not relevant. For D_{eR} values ranging between 10 and 25% of the total dose (area 2), the sediments are considered as potentially datable but such dating application is hazardous. According to the transport condition, the corresponding time zone is comprised between about 15 and 75 ka.

Three other zones (3, 4 and 5) have been defined, in which the ages should be more reliable because the D_{eR} represents then between 5 and 10% of the total dose for the first, between 1 and 5% for the second and less than 1% for the last one.

All of these results show that, even if bleaching is not complete at the deposition time for fluvial, marine or aeolian sediments, the observed low values of the resulting residual Dose does not prohibit dating these sediments by ESR. Nevertheless, as a precaution, it is not recommended to work on samples whose age would be younger than 75 ka.

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