Received: 12 September 2016 | Revised: 12 January 2017 | Accepted: 15 January 2017

DOI 10.1002/ajpa.23180

TECHNICAL NOTE

WILEY American Journal of PHYSICAL ANTHROPOLOGY The Official Journal of the American Association of Physical Anthropologists

Quantifying the impact of μCT-scanning of human fossil teeth on ESR age results

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Funding Information
The research leading to these results has received funding from the People Programme (Marie Curie Actions) of the European Union's Seventh Framework Programme (FP7/2007–2013) under REA Grant Agreement ~~no~~ PIOF-GA-2013-626474. Some aspects of this work have also been covered by an Australian Research Council Future Fellowship grant (FT150100215).

Abstract

Fossil human teeth are nowadays systematically CT-scanned by palaeoanthropologists prior to any further analysis. It has been recently demonstrated that this noninvasive technique has, in most cases, virtually no influence on ancient DNA preservation. However, it may have nevertheless an impact on other techniques, like Electron Spin Resonance (ESR) dating, by artificially ageing the apparent age of the sample. To evaluate this impact, we μCT-scanned several modern enamel fragments following the standard analytical procedures employed by the Dental Anthropology Group at CENIEH, Spain, and then performed ESR dose reconstruction for each of them. The results of our experiment demonstrate that the systematic high-resolution μCT-scanning of fossil hominin remains ~~introduceing~~ a nonnegligible X-ray dose ~~to~~ the tooth enamel, equivalent to 15–30 Gy depending on the parameters used. This dose may be multiplied by a factor of ~8 if no metallic filter is used. However, this dose estimate cannot be universally extrapolated to any μCT-scan experiment but has instead to be specifically assessed for each device and set of parameters employed. The impact on the ESR age results is directly dependent on the magnitude of the geological dose measured in fossil enamel but could potentially lead to an age overestimation up to 40% in case of Late Pleistocene samples if not taken into consideration.

KEYWORDS

μCT-scan, electron spin resonance dating, fossil tooth enamel, X-ray dose

1 | INTRODUCTION

The development of noninvasive techniques has always been crucial in palaeoanthropological studies in order to obtain key information from invaluable bone and dental remains without causing visible damages. In that regard, conventional X-ray computed (micro-)tomography ([μ]CT) has become an increasingly popular tool over the last decade, as it enables to produce high resolution 3D images of human fossils and allows access to the internal structures of remains without any physical interference (e. g., Martín-Francés, Martinon-Torres, Gracia-Téllez, & Bermúdez de Castro, 2015; Olejniczak et al., 2008). This is why systematic μCT-scanning of fossil human teeth or bones prior to any further analysis is now routinely performed by most palaeoanthropological research teams around the world. However, although noninvasive, X-ray irradiation may nevertheless impact the fossil remains at different levels. For example, Immel et al. (2016) recently assessed whether μCT-scan analysis could damage ancient DNA remains in fossil bones. Their results showed that no significant effect could be detected for doses below 200 Gy, a level that most conventional μCT instruments usually do not reach. However, even below this threshold such dose estimates suggest that those analyses may have potentially a non-negligible impact on dating studies carried out by electron spin resonance (ESR). Indeed, with the recent development of semidestructive approaches combining ESR measurements of enamel fragments and U-series analyses by laser ablation ICP-MS, valuable fossil human teeth are being increasingly dated using this method (e. g., Grün et al., 2006; Torres et al., 2010). The ESR method is by definition based on the quantification of the effects of ionizing radiations onto fossil enamel. Those radiations are usually coming from natural sources, either from the radionuclides present in the tooth itself or in the surrounding environment (see an overview in Duval, 2015). Consequently, X-ray imaging may potentially induce an additional dose to that already absorbed by the fossil enamel over time (geological dose), leading to an age overestimation if not taken into consideration. A previous study by

TABLE 1 µCT-scan parameters used in the three different configurations depending on the degree of sample fossilization

Sample	#1	#2	#3
Parameters	Modern human (MH)	Sima de los Huesos (SH)	Gran Dolina-TD6 (GD)
Voxel size (isometric) (mm)	0.018	0.018	0.018
Voltage (kV)	100	120	140
Amperage (µA)	100	110	120
Filter	2 mm x 0.1 mm copper	2 mm x 0.1 mm copper	2 mm x 0.1 mm copper
Number of images	1,800	1,800	1,800
Average	5	5	5
Skip	3	3	3

Grün, Athreya, Raj, and Patnaik (2012) suggested that high resolution µCT-scanning may produce a significant dose of several hundreds of Gy in the tooth enamel. If confirmed, such values may simply preclude any further ESR dating of remains that have been previously µCT-scanned. However, it is also known that many sources of uncertainties may impact, to a greater or lesser extent, the magnitude of this laboratory dose: it may strongly vary (by a factor of >300 according to Figures 7 and 8 from Immel et al., 2016) depending on the device used, the characteristic of the X-ray source or the acquisition parameters, such as the scanning time, the position of the tooth with respect to the X-ray source, or even the nature of the sample holder (e. g., Grün, Mahat, & Joannes-Boyau, 2012).

Over the recent years, the Dental Anthropology Group (GAD) at the Centro Nacional de Investigación sobre la Evolución Humana (Burgos, Spain) has been systematically µCT-scanning Atapuerca fossils following the procedure described in Martínez de Pinillos et al. (2014) and Martinón-Torres et al. (2011). Although it may be expected in first instance that this routine may produce a non-negligible X-rays dose into the enamel, the magnitude of this dose is for the moment simply unknown. In addition, this dose may vary depending on the fossil sample considered and its degree of fossilization. Indeed, for obtaining optimum images (i. e., with good contrast resolution and minimum artefacts) in denser materials it is necessary to adjust the scan parameters, which usually leads to deliver higher radiation doses.

Given this uncertainty, we carried out a series of µCT-scans experiments on several modern enamel fragments using the standard procedures developed and routinely used by the GAD, in order to assess the potential impact, if any, of those analyses on the age results that might be derived from subsequent ESR dating.

2 | MATERIALS AND METHODS

2.1 | µCT-scan analysis

Based on our experience in µCT-scanning fossil teeth, we observed that depending on the conservation state (degree of fossilization) of each sample, it is necessary to adjust the acquisition parameters in order to achieve satisfying image results. Nevertheless, these are at the same time usually almost constant for each of dental human collections, that is, for various fossils from a given site or samples with similar chronologies. Samples from modern and fossil collections may usually be classified in three different categories, from low to high degree of fossilization, corresponding to different sets of parameters with voltage and amperage ranging from 100 to 140 kV and 100 and 140 µA, respectively (e. g., Martínez de Pinillos et al., 2014; Martinón-Torres et al., 2011).

To carry out the present study we selected two modern (most likely present day) bovid teeth (MOD1601 and MOD1602) because: (a) the dose naturally absorbed by modern teeth is usually very small and does not exceed a few Gy contrary to fossil teeth, and in first instance should thus not interfere with the dose given by µCT scan analyses; (b) modern tooth enamel is known to be an excellent ESR dosimeter, as it can accurately register very small dose values (<1 Gy) and shows an excellent response to the dose (Fattibene & Callens, 2010 and references therein); and (c) the radiation sensitivity of bovid tooth enamel was found to be close to that of human teeth (Toyoda et al., 2003). Those two bovid teeth are surface finds from a pasture in Northern France, so their exact provenience is unknown, as well as their dosimetric history or approximated age.

Four enamel fragments of ~300–400 mg (labeled #1–4) were extracted from MOD1601. Three of these fragments (#1–3) were µCT-scanned at CENIEH with a GE Phoenix v/tome/x s 240 instrument and following the standard GAD protocol (e. g., Martínez de Pinillos et al., 2014; Martinón-Torres et al., 2011). A specific set of parameters was applied to each sample, that is, corresponding to those usually employed for modern human (MH), Sima de los Huesos (SH; i. e., Middle to Late Pleistocene samples) and Gran Dolina-TD6 (GD; i. e., Early Pleistocene samples) teeth. The parameters are listed in Table 1. Total scanning time was approximately 52 minutes per sample. The fourth fragment (#4) was not µCT-scanned in order to evaluate the background dose naturally present in this bovid tooth.

The sample holder was designed to accommodate all samples, avoiding misplacement or changes of the position. It was attached to the µCT sample holder at the beginning and its position did not change afterwards (during the three scans). The resolution was the same for the samples (0.018mm). The three samples were placed with the buccal surface facing the X-ray source.

COLOR

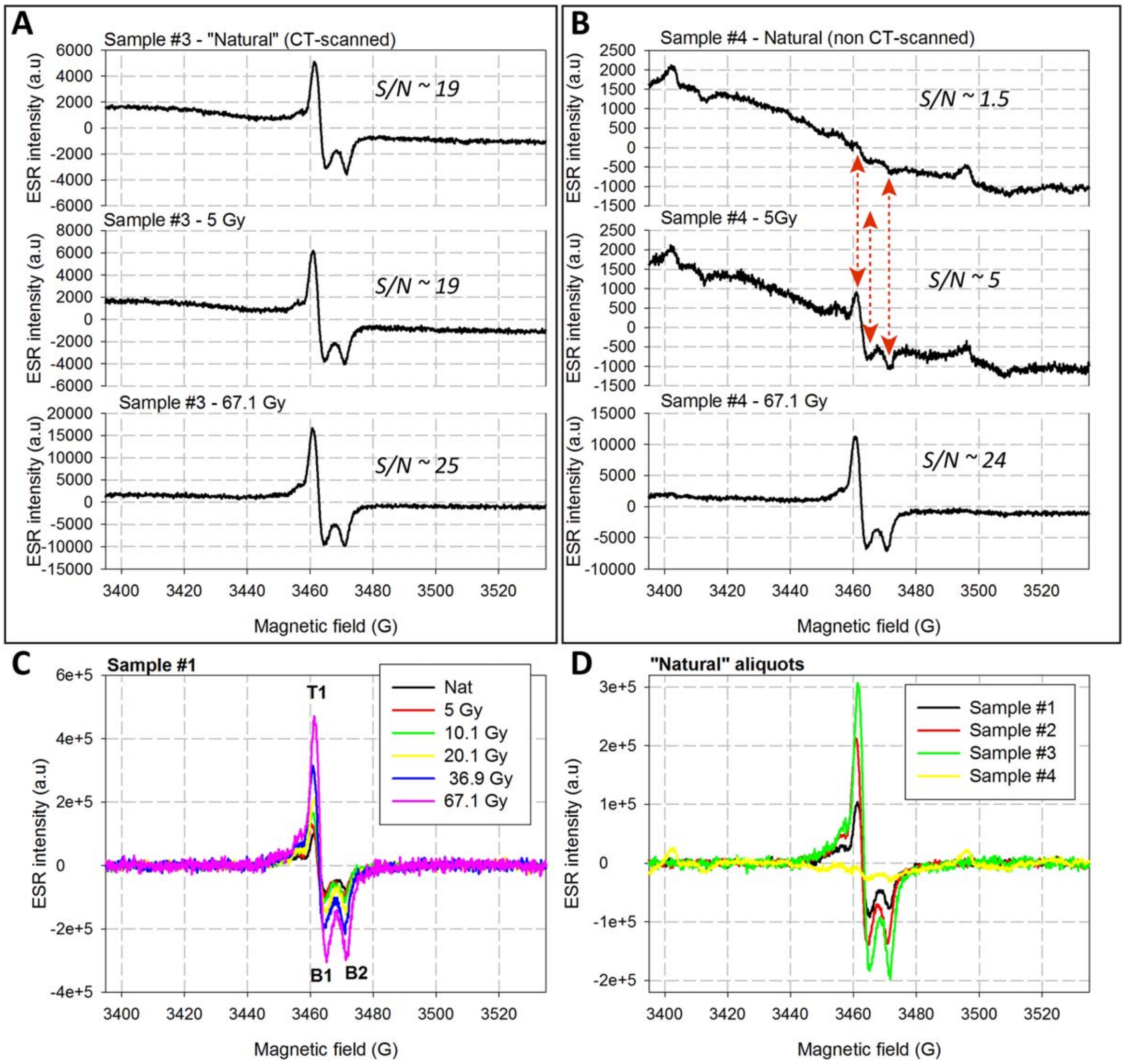

FIGURE 1 Examples of ESR spectra (normalized to 1 scan and 1 mg) obtained for the present study, without (A and B) and with (C and D) baseline correction. ESR intensities were extracted by peak-to-peak measurements between T1 and B2 after baseline correction (C). Estimated signal-to-noise (S/N) value (done after baseline correction) is also indicated on spectra of (A) and (B). (B) The arrows indicate the position of the radiation induced ESR signal in the non-CT scanned aliquot of sample #4. (C) ESR spectra corresponding to sample #1. (D) ESR spectra corresponding to the first aliquot of each sample (i. e., the μCT-scanned aliquots for samples #1 to #3 and the natural one for #4) [Color figure can be viewed at wileyonlinelibrary.com]

Another CT-scan experiment was then performed with the second tooth MOD1602 in order to evaluate the impact of use of a metallic filter on the dose absorbed by the samples. Two enamel fragments (labeled #5 and #6) were extracted. Fragment #5 was μCT-scanned with the SH parameter set (like #2) but without metallic filter. Fragment #6 was not μCT-scanned in order to evaluate the background dose naturally present in MOD1602.

2.2 | ESR dosimetry

In this study, dose evaluations are performed following the standard procedure used in ESR dosimetry/dating, that is, via the Multiple Aliquot Additive dose method (Duval, 2015), in order to obtain results that are directly comparable with those derived from the ESR dating studies. Once μCT-scanned, the fragments are powdered and then gamma irradiated. The average X-ray dose absorbed by the volume of each fragment is thus expressed in terms of gamma equivalent dose (D_E). This methodology is similar to that employed by Grün, Athreya, et al. (2012), while the dosimetry evaluation in Immel et al. (2016) were obtained with a dosimeter equipped with a ionization chamber producing water surface equivalent X-ray dose estimates.

ESR dose reconstruction was performed at the CENIEH (Burgos, Spain) following a standard procedure similar to that described in Duval et al. (2013). The enamel fragments were powdered and sieved <200 μm. Each sample of tooth MOD1601 (#1–4) and MOD1602 (#5 and #6) was divided into several aliquots and gamma-irradiated with a Gammacell-1000 Cs-137 source. The following doses were given to the 6 aliquots of samples #1–4: 0, 5.0, 10.1, 20.1, 36.9, and 67.1 Gy.

COLOR

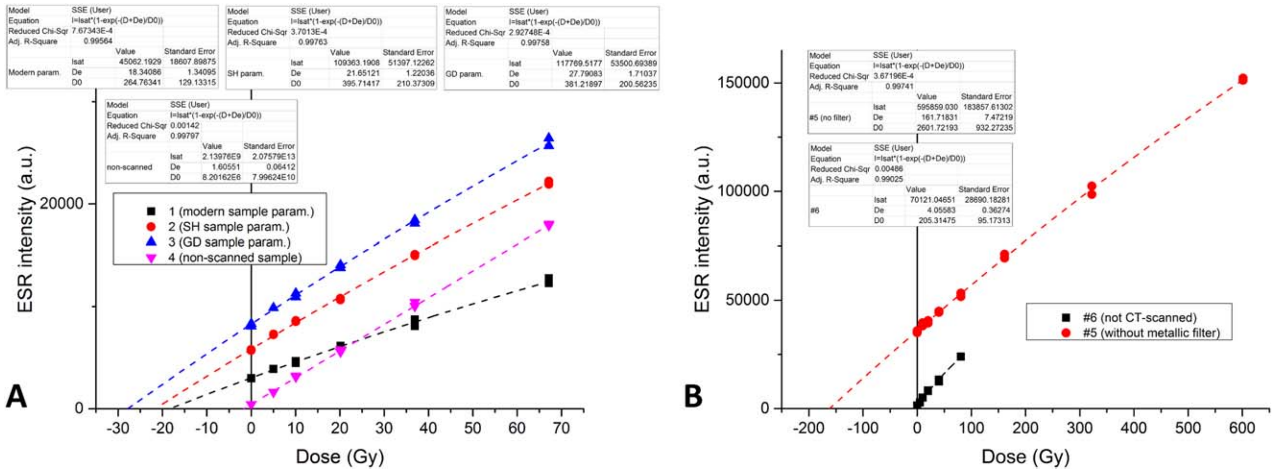

FIGURE 2 ESR dose–response curves (DRCs). Key: (A) First experiment carried out on samples #1 to #4 and (B) second experiment carried out on samples #5 and #6. The intersection between the DRCs of samples #1 and #4 may be in first instance surprising, but it might be simply due to a slight difference in the radiation sensitivity of the two samples. Although they were both taken from the same tooth, each fragment was collected from a slightly different area of the tooth, which may show different crystalline characteristics generating slightly different responses to the dose due to variable proportions of the different types of CO_2^- radicals (Joannes-Boyau & Grün, 2011). This difference, however, does not affect the D_E estimates, which follow instead a logical increasing pattern (4 < #1 < #2 < #3) [Color figure can be viewed at wileyonlinelibrary.com]

Samples #5 was irradiated as follows (eight aliquots): 0, 10.0, 20.0, 40.1, 80.6, 161, 322, and 601 Gy. Finally, the six aliquots of sample #6 (not µCT-scanned) were given: 0, 5.0, 10.1, 20.1, 40.1, and 80.6 Gy.

ESR measurements were carried out at room temperature with an EMXmicro 6/1 Bruker ESR spectrometer coupled to a standard rectangular ER 4102ST cavity. In order to minimize the uncertainty on the measurements, the analytical procedure was standardized as follows. First, all the aliquots of a given sample were carefully weighted into their corresponding tubes, and a maximum deviation of 1 mg was tolerated from one aliquot to another. ESR measurements were performed using a Teflon sample tube holder inserted from the bottom of the cavity. Although this device may slightly decrease the cavity sensibility, it ensures that the vertical position of the tubes remains exactly the same for all aliquots. The following acquisition parameters were used: 5–600 scans (depending on the sample and aliquot measured), 1 mW microwave power, 1,024 points resolution, 15 mT sweep width, 100 kHz modulation frequency, 0.1 mT modulation amplitude, 20 ms conversion time, and 5 ms time constant.

Because some of the spectra were showing a non-horizontal baseline given the low ESR intensities of the signals recorded (Figure 1A and B), it has been decided to proceed to a baseline correction using the Bruker WINEPR System (v.2.22) software. The following procedure was systematically applied to all aliquots: regions of the spectrum without signal at low and high magnetic field values (<3,420 and >3,510 G) were defined to calculate a baseline based on a fifth order polynomial function, which was then subtracted to the main signal. Some examples of the resulting spectra may be seen in Figure 1C and D. Further detail about the baseline correction procedure can be found in the Bruker WINEPR System User manual. ESR intensities were extracted from T1-B2 peak-to-peak amplitudes of the ESR signal of enamel (Figure 1C), and then corrected by the corresponding number of scans and aliquot mass. Signal-to-noise (S/N) estimates are obtained by dividing the intensities of the ESR signal by that of the noise (IAEA, 2002). The latter is evaluated by measuring the maximum peak-to-peak amplitude in a high magnetic field domain (e. g., >3,510 G, Figure 1) where no signal is observed. It should be noted that S/N values are systematically ≥5, except for the natural aliquot of samples #4 and #6 for which S/N ≈ 1.5 and 4, respectively.

Fitting procedures were carried out with the Microcal OriginPro 9.5 software using a Levenberg-Marquardt algorithm by chi-square minimization. D_E values were obtained by fitting a single saturating exponential (SSE) function through the experimental data, with data weighting by the inverse of the squared ESR intensity ($1/I^2$) (Duval, Grün, Falguères, Bahain, & Dolo, 2009).

Each sample was measured two times in order to evaluate measurement and D_E repeatability. For all teeth, the two times provided consistent dose results, that is, within error. Consequently, final D_E values were calculated for each sample by pooling all the ESR intensities derived from the repeated measurements in a single DRC (Duval, Guilarte Moreno, & Grün, 2013) (Figure 2).

3 | RESULTS

Goodness-of-fit is excellent (adjusted r^2 systematically >0.99), indicating thus the reliability of the fitting results (Duval et al., 2013). Dose results obtained for samples #1–3 vary within relatively narrow range, between 18.3 ± 1.34 and 27.8 ± 1.71 Gy (Figure 2), but show nevertheless an interesting pattern. Basically, the procedure routinely used for modern samples (MH) produces a dose in the enamel that is slightly, but significantly, lower to that used for the fossilized samples. In comparison, the GD analytical procedure introduces a dose that is higher to that of SH experimental setup (27.8 vs. 21.7 Gy, respectively). Such a

TABLE 2 Overview of the dose results obtained in the present study

	D_E (Gy)		Effective dose* (Gy)	
	With baseline correction of the signal	No baseline correction of the signal	(a) Dose subtraction	(b) ESR intensity subtraction
Sample #1	18.3 ± 1.3	20.4 ± 1.3	16.7 ± 1.3	13.6 ± 0.9
Sample #2	21.7 ± 1.2	23.6 ± 1.5	20.0 ± 1.2	18.4 ± 1.0
Sample #3	27.8 ± 1.7	29.2 ± 2.1	26.2 ± 1.7	24.8 ± 1.5
Sample #4	1.6 ± 0.1	3.~~15~~ ± 0.2	–	–
Sample #5	161.7 ± 7.5	166.7 ± 7.7	157.7 ± 7.5	154.3 ~~±~~ 7.2
Sample #6	4.1 ± 0.4	5.6 ± 0.4	–	–

Key (*): the effective dose corresponds to the dose given by the µCT scan analysis: it is calculated by subtracting the natural (background) dose present in the modern bovid tooth before the experiment to the D_E assessed after the µCT scan analysis. Two ways of calculations are explored: (a) by subtracting the D_E value obtained for the ~~nonscanned~~ samples (#4 and #6) and (b) by subtracting the ESR intensity of the non-scanned aliquot to the ESR intensities of all the other aliquots (similarly to the procedure employed for removing the residual ESR intensity of the Al signal in ESR dating of quartz; e. g., Voinchet et al., 2003).

pattern could actually be expected, given the different amperage and voltage values used for each procedure (MH < SH < GD; Table 1).

For comparison, sample #4 (~~nonscanned~~ fragment) shows a slight dose of ~1.6 Gy (Figure 2). This dose result should be removed to that of the other three samples in order to obtain the effective dose values given by X-ray imaging, resulting in 16.7 ± 1.3~~4~~ Gy (#1), 20.0 ± 1.2~~2~~ Gy (#2), and 26.2 ± 1.7~~1~~ Gy (#3) for MH, SH, and GD analytical procedures, respectively.

The second µCT-scan experiment carried out using SH experimental setup but without the metallic filter provides a significant dose results of 162 ± 7.5 Gy. When removing the background dose of 4.1 ± 0.~~36~~ Gy given by sample #6, the dose effectively absorbed by enamel sample #5 during this experiment is of 158 ± 7.5 Gy. This value is about eight times higher to that evaluated for sample #2 analyzed in the same conditions, but with metallic filter, demonstrating thus (as it could be expected) the significant impact of this parameter on the dose absorbed by the tooth. An overview of the dose results is displayed in Table 2.

4 | DISCUSSION

4.1 | Impact of baseline correction on the ESR results

Due to the weak ESR intensities measured for most of the aliquots (and especially for the less irradiated ones), a nonhorizontal baseline may be observed on some spectra (see Figure 1A and B). Consequently, a correction appeared to be necessary in order to avoid any significant overestimation in the evaluation of the intensities. Such procedure is usually not necessary in ESR dating of fossil teeth, as the fossil teeth have in almost all cases a dose of at least several tens of Gy. ~~For example,~~ this baseline correction has a small impact on the ESR intensities of sample #3, which decrease between 2 and 4% from the most irradiated aliquot to the first one. As a consequence, the resulting D_E is 6% lower than that would have been obtained without baseline corrections, but both D_E values are nevertheless within 1σ error. For all the samples presenting a dose between 10 and 30 Gy (#1–3), the absence of baseline correction would induce a dose overestimated by about 2 Gy, while this reaches 5 Gy for sample ~~with a~~ D_E of 160 Gy ~~(#5)~~. Despite this systematic bias, it is nevertheless worth mentioning that all values remains within error (see Table 2). The major impact is found for the two non µCT-scanned samples showing a D_E value <10 Gy. For example, sample #4: the baseline correction induces a decrease of the ESR intensity of only 2% for the most irradiated aliquot at 67.2 Gy, but by about −50% for the first point of the DRC. Consequently, the resulting D_E considering baseline corrections is 50% lower than if no corrections had been performed.

These results illustrate the importance of carrying out this baseline correction prior to the evaluation of the ESR intensities for the present data set. If for most of the aliquots it has a very limited impact on the ESR intensities (<5% for any aliquot irradiated to >20 Gy), the absence of baseline correction would automatically induce a dose overestimation, whose significance would be inversely proportional to the amount of dose previously absorbed by the sample.

4.2 | Impact of the natural (background) dose on the final effective dose estimates

The D_E estimate initially calculated for sample #4 (~~nonscanned~~) might be in first instance considered as a maximum possible estimate. Indeed, the small radiation-induced ESR signal intensity measured in the natural (nongamma irradiated) aliquot (Figure 1B) of sample #4 might be slightly overestimated, as it is actually quite close to background levels (S/N = 1.5). When subtracting the noise intensity to that of the signal of all aliquots from sample #4, the resulting D_E is of 0.56 ± 0.03 Gy, that is, ~1/3 of the previous estimate (1.6~~0~~ ± 0.~~06~~ Gy). It should be noted here that the other samples are not affected in the same extent by the noise, as the resulting D_E estimates after noise subtraction remain within 1σ error.

The presence of some interfering native signals at high magnetic field values (>3,500 G) for the spectra associated to low irradiation dose values (and especially the natural aliquot of #4) may interfere with the evaluation of the noise intensity and most likely result in a

TABLE 3 Potential relative weight of the X-ray dose into the total (geological) dose calculated for those samples by ESR in case these samples had been previously CT-scanned at CENIEH following the GAD procedure

Site	Fossil remain	ESR age	Published equivalent dose (Gy)	Potential impact (%)
El Sidrón (Asturias)	Neanderthal isolated incisor	38.5 ± 4.5 ka (Torres et al., 2010)	32.3 ± 1.1	38
Banyoles (Catalunya)	Human tooth	66 ± 7 ka (Grün et al, 2006)	155 ± 0.6	11
Atapuerca Sima de los huesos	Bear tooth	261 ± 26 25 ka (Arsuaga et al., 2014)	444 ± 22	4
Mauer (Germany)	Herbivorous tooth (M0507)	624 ± 79 73 ka (Wagner et al., 2010)	813.8 ± 18.5	3.1
Atapuerca Gran Dolina	Equid tooth (AT9603)	770 ± 116 ka (Falguères et al., 1999)	846 ± 56	3.0

For samples from El Sidrón, Banyoles, and Sima de los Huesos, an effective dose of 20.0 Gy was considered, while a value of 26.2 Gy was used for the older, and more probably more fossilized, Mauer and Gran Dolina samples.

somewhat overestimated value (Figure 1B). Consequently, the true background dose absorbed by the modern tooth in nature since the death of the animal is more likely somewhere between ~0.6 and ~1.6 Gy. Given this uncertainty, one should keep in mind the possibility that the effective dose values previously calculated (i. e., 16.7 ± 1.34, 20.0 ± 1.22, and 26.2 ± 1.71 Gy for MH, SH, and GD analytical procedures, respectively) might be slightly underestimated by <1 Gy.

Finally, the background dose derived from sample #6 is somewhat higher to that obtained from #4 (Table 2), but it represents only 2.5% of the total dose measured for sample #5. Consequently, it has a very limited impact on the effective dose, which remains within 1σ error with the D_E result.

4.3 | Evaluation of the effective dose by subtracting the natural ESR signal

Effective dose values were initially calculated by subtracting the D_E calculated for the non-µCT-scanned samples (i. e., #4 and #6) (see Table 2). However, it should also be possible to assess the effective dose by considering the ESR intensity of the natural aliquot of #4 and #6 as a residual ESR intensity. The natural ESR intensity may be subtracted from the ESR intensities of all the other aliquots, similarly to the procedure used to remove the unbleachable component of the ESR signal of the Aluminum center in ESR dating of quartz grains (e. g., Voinchet et al., 2003). To test this procedure, the "natural aliquots" of samples #1–4 were measured together with the same experimental conditions in order to avoid any bias that would preclude their direct comparison: the ESR intensity of #4 was found to be 0.26, 0.15, and 0.11 of that of the natural aliquots of samples #1–3, respectively. This relative natural ESR intensity was then subtracted from the ESR intensities of all aliquots of #1–3, and new D_E values were obtained (Table 2, last column). Similarly, a ratio of 0.04 was found for sample #6 in comparison with the natural ESR intensity of #5, and a new D_E value was derived for #5. This approach produces effective dose values that are systematically lower by about 2 Gy in comparison with the dose subtraction approach. However, the effective dose values derived from the two procedures are nevertheless all in close agreement, consistent at 1σ, except for sample #1 (2σ). Given the low S/N observed in the natural aliquot of samples #4 and #6, we would nevertheless consider in first instance that effective dose estimates based on dose subtraction may be somewhat more reliable, as the results do not rely only on a single aliquot showing a very weak ESR signal.

4.4 | Comparison with previous works

The experimental results illustrate the variability of dose that may be given to tooth enamel during µCT-scan analyses. As expected, dose values are clearly parameter dependent, which makes comparisons with previous studies by Grün, Athreya, et al. (2012), Grün, Mahat, et al. (2012) and Immel et al. (2016) not so straightforward. Following a somewhat similar methodology to that of the present work, Grün, Athreya, et al. (2012) nevertheless estimated the dose values given by CT-scanning to be between 250 and 420 Gy, that is, more than 10 times higher than our estimates. If the dose difference may be partially explained by a distinct experimental setup (e. g., 180 kV, 0.11 mA current; BIR ACTIS CT scanner), the authors do not mention the use of a metallic filter during scanning. This may actually be the main source of difference between the two studies, as our results show that the dose absorbed by the enamel is about eight times lower when using a metallic filter (Table 2). This is consistent with previous observations by Immel et al. (2016) (see Figure 8 of their work): these authors indicate that for a given device and configuration the use of a filter may divide the dose absorbed by the enamel by a factor of >6.

Other additional factors may possibly explain, at least partially, the differences with the results obtained by Grün, Athreya, et al. (2012), such as the positioning of the tooth with respect to the X-ray source, or even the nature of the sample holder (e. g., Grün, Mahat, et al., 2012). However, given the number of sources of uncertainty that may influence the dose effectively absorbed by the tooth, it seems to us that any further explanation would be quite speculative.

4.5 | Potential impact on ESR age results

Our results indicate that the systematic μCT-scanning of fossil remains following GAD scanning protocol introduces effective dose values into the enamel that are ranging somewhere between 15 and 30 Gy depending on the acquisition parameters selected. Although those values may seem in first instance small, they definitely have a nonnegligible impact on the ESR age results if not removed. This impact will be greater if the ESR equivalent dose gets smaller (or the sample gets younger). To illustrate this, Table 3 presents an overview of recent ESR dating studies carried out on either human or animal teeth from various Early to Late Pleistocene palaeoanthropological sites. Depending on their chronology, a laboratory dose of 20.0 or 26.2 Gy would have been added to the geological dose measured if those samples had been μCT-scanned at CENIEH following the GAD procedure. This would have corresponded to a nonnegligible dose overestimation, and thus an age overestimation, ranging from ∼38% for a sample from El Sidrón to ∼3.0% for an Atapuerca Gran Dolina tooth. Consequently, these results demonstrate the necessity to accurately evaluate the dose given to the enamel by μCT-scanning in case subsequent ESR dating is planned.

5 | CONCLUSION

The results of this experiment show that the systematic high resolution μCT-scanning of fossil hominin remains introduces a nonnegligible X-ray dose to the tooth enamel, and especially if no metallic filter is used. The impact on the ESR age results is directly dependent on the magnitude of the geological dose absorbed by the sample, but could potentially lead to an age overestimation up to 40% in case of Late Pleistocene samples.

The laboratory X-ray dose is strongly device and procedure dependent, and the estimate obtained in this work cannot be universally used. It is rather specific to the GAD analytical procedure used in combination with the GE Phoenix v/tome/x s 240 instrument at CENIEH.

Although it is recommended in first instance to avoid any previous CT-scanning of fossil remains if the sample is intended to be dated by ESR, we understand this may not be always possible given the value of those remains. Therefore, we recommend scanning a modern tooth together with the human fossil using the same device and acquisition parameters, in order to obtain a fair estimation of the X-ray dose given to the fossil sample ~~that~~, could then be subtracted from the ~~geological dose~~.

ACKNOWLEDGMENTS

The research leading to these results has received funding from the People Programme (Marie Curie Actions) of the European Union's Seventh Framework Programme (FP7/2007–2013) under REA Grant Agreement no PIOF-GA-2013-626474. Some aspects of this work have also been covered by an Australian Research Council Future Fellowship grant (FT150100215). LM-F is beneficiary of a Fundación Atapuerca Post-Doctoral Research Grant. The authors would like to thank M. Lebon (MNHN) for supplying the modern bovid teeth and M. Martinón-Torres (UCL) and J.M. Bermúdez de Castro (CENIEH) for their support and useful advice on the manuscript. Two anonymous reviewers provided constructive comments on an earlier version of the article.

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How to cite this article: Duval M, Martín-Francés L. Quantifying the impact of μCT-scanning of human fossil teeth on ESR age results. *Am J Phys Anthropol*. 2017;00:1–8. https://doi.org/10.1002/ajpa.23180