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## Densities of Modern and Fossil Dental Tissues: Significance to ESR Dating of Tooth Enamel

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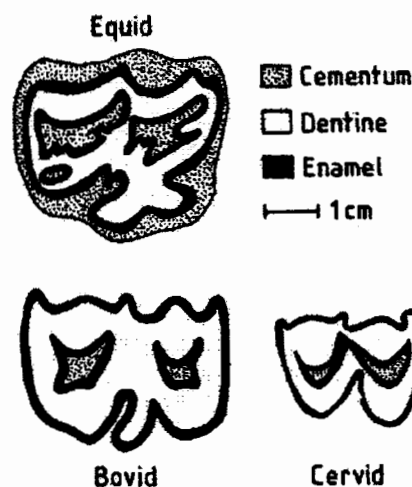
**Abstract.** We measured the density of enamel, dentine and cementum from teeth that range in age from 0-450 ka. We found that the density of dentine and cementum is significantly higher in samples of Palaeolithic age or older relative to young archaeological teeth and modern teeth from an abattoir. These density changes occur within the first 50,000 years of burial. This density change is important for new calculations of beta attenuation in tooth enamel based on One-Group Theory, which requires density input data in order to calculate energy transport across interfaces between enamel and adjacent sediment, cementum or dentine. Enamel densities were unchanged over the time interval between 0 and 450 ka.

### Introduction

Electron spin resonance (ESR) dating of tooth enamel has now been used to establish the ages of many Palaeolithic archaeological sites as well as sites of importance to Quaternary geology (Rink, 1997). Though still experimental in some regards, the technique as originally proposed by Grün et al. (1987) is still in use, and has shown good agreement with other independent dating methods (Rink et al., 1996a, Monigal et al., 1998).

Horse, cow and deer teeth are characterized by layered arrangements of three different dental tissues: cementum, dentine and enamel (Fig. 1). In horse molars, the outer layer of cementum can be quite thick (2-3 mm) near the top of the tooth (chewing end), and generally thins to less than 0.5 mm toward the base of the tooth. The same is true for teeth of wild ass (*Equus hydruntinus*). The outer cementum layer on fossil cow and deer teeth is rarely as well preserved as it is in horse and wild ass teeth, but thicknesses approaching that seen in modern cow samples (up to 0.5 - 1 mm) are seen in some exceptional cases, and also in cases where deer teeth are heavily worn. It is usually easy to avoid areas covered with thin layers of fossil cementum on cow teeth, but this is more difficult in cases where relatively thinner layers occur on well-preserved, unworn cervid teeth. For cases where enamel, dentine or cementum are strong beta dose sources, or where a large fraction of the total dose is transmitted from sediment through a low U-content

cementum layer, the ESR ages are strongly dependent upon calculations of beta dose attenuation in teeth.



**Figure 1.**

*Occlusal surface view (chewing surface) of fossil molar teeth commonly used for ESR dating. The outer enamel in equid (horse) teeth is covered by cementum, while in bovid (cow) and cervid (deer) teeth, the outer enamel layer is exposed to sediment at positions near the occlusal surface (in some cases thin layers of cementum are preserved on cervid and bovid teeth).*

Beta particles are strongly attenuated by dental tissues. Beta attenuation in tooth enamel must be accounted for in ESR dating (Grün, 1986). The

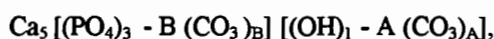
gradient of beta attenuation in the enamel depends, amongst other things, on the density of the enamel.

The fraction of beta dose emitted by dentine is also controlled by the density-dependent self-absorption of the beta particles in the dentine. Moreover, in some teeth enamel is partly covered by a layer of cementum (up to 3 mm thick) which may act both as a source of beta rays and as an absorber of beta radiation during transmission of sediment-sourced beta radiation. The beta attenuation is dependent on the density of the cementum. For all these reasons it is necessary to know the density of cementum, dentine and enamel, and the present work was dedicated to determining densities of these tissues.

Since 1987, the McMaster ESR dating group has been using the computer software DATA (provided graciously by R. Grün of Australian National University) to calculate ESR dates. This program assumes that infinite matrix beta doses from uranium in the cementum and dentine elements are absorbed by the enamel. In order to use this software, the actual layers of cementum and dentine on the tooth must be about 2 mm thick, which satisfies the assumption that these dose sources are an infinite matrix with respect to the maximum range of the beta particles (about 2 mm). In this approach, ages calculated by DATA are assumed to be independent of the actual density of the cementum and dentine. However, many teeth have cementum and dentine layers which are too thin to satisfy the infinite matrix assumption. At McMaster Geology, we developed an ESR dating software called ROSY, which allows us to date teeth with any thickness of dentine or cementum (Brennan *et al.*, 1997), now that we have measured their densities (and that of enamel) in fossil teeth. This program utilizes One-Group Theory (O'Brien *et al.*, 1964, Prestwich *et al.*, 1997) to model the beta radiation fluxes across the boundaries between sediment, cementum, enamel and dentine. We can incorporate a value for the fractional water content of these materials, which of course modifies their density in the actual calculation. The One-Group beta attenuation calculation yields considerably lower beta doses to enamel (Brennan *et al.*, 1997) than the beta attenuation calculation of Grün (1986).

Gilda (1951) reports densities for rat and

hamster dental tissues, but we are not aware of reported densities for horse or cow dental tissues. Differences in density among the dental tissues of a given species are related to their degree of mineralization (Fig. 2). The mineral phase of these materials is widely regarded to be the mineral dahllite (Lowenstam and Weiner, 1989) which is a carbonate hydroxyapatite with the formula



where the carbonate can substitute at either the  $\text{PO}_4$  site (B) or the OH site (A).

### Experimental Methods and Samples

Table 1 lists the origin, taxa and approximate burial age of the samples we have used. Enamel, dentine and cementum samples were separated from one another using a dental drill followed by visual inspection at 15-40 x magnification to insure purity.

Sample density,  $D_s$ , determinations were made using a pycnometer flask, electronic balance and the following equation:

$$D_s = \{M_s / [(M_1 - M_2) - (M_3 - M_4)]\} \{D_{\text{isop}}/D_{\text{water}}\} \quad (1)$$

where  $M_s$  is the dry sample mass,  $M_1$  is the mass of the flask filled with isopropyl alcohol,  $M_2$  is the mass of the flask,  $M_3$  is the mass of the flask filled with isopropyl alcohol and containing the sample,  $M_4$  is the mass of the flask plus the dry sample,  $D_{\text{isop}}$  is the density of isopropyl alcohol (0.785) and  $D_{\text{water}}$  is the density of water (water was used as the fluid for some of the enamels, thus the last term of the equation was not used). This calculation is based on the buoyancy of the sample in a fluid (Archimedes principle), where the sample density is given by its (weight in air)/(weight in air - weight in fluid of known density).

Bubbling occurred for tens of minutes during the wetting of the cementum and dentine, as isopropyl alcohol filled the pore spaces. Measurements had to wait until bubbling ceased. Replicate analyses were made by partial emptying and refilling of the flask with isopropyl alcohol, but using the same dry weight for the sample (the sample was wet with isopropyl only one time).

The values that have been measured are the pure mineral density (without pore space) based on the assumption that the bubbling has caused all of

the pore space to be filled with fluid before the density value was determined. For the purposes of the age calculation by ROSY, this value is the "dry density" of the tissue, which is modified by an additional input value for the fractional water content, which is used to modify the dry density for the beta dose calculation.

Accuracy of the pycnometer was checked using a quartz density standard. The expected values are 2.65 - 2.67 g cm<sup>-3</sup> for quartz. We interspersed measurements of the standard with those of the dental tissues. For the three enamels measured in water (92050, 91182 and 2060), we measured the quartz standard 11 times on the same day as all three enamel densities were determined. The value was 2.69 +/- .03 (1s). All of the remaining samples were measured in isopropyl alcohol over a period of 27 days. During that time the quartz standard was measured a total of 5 times on three different measurement days. This yielded a value of 2.66 +/- .03 (1s).

### Results

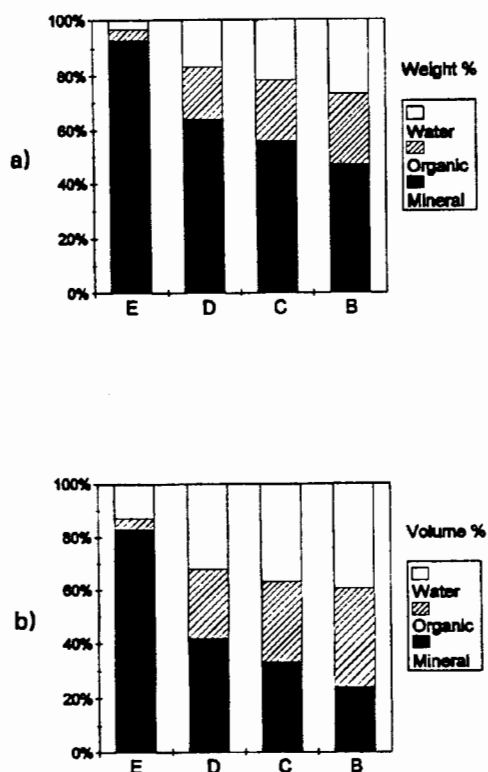
The density values of enamel, dentine and cementum are given in Table 1. Our values for relatively modern cementum and dentine are slightly higher than, but similar to densities of human cementum and dentine of 2.03 and 2.14 g cm<sup>-3</sup> respectively (Rowles 1967). Enamel values are in excellent agreement with published values of 2.9-3.0 g cm<sup>-3</sup> for human enamel (Rowles, 1967). Table 1 shows that consistently higher values for fossil cementum and dentine were found relative to modern or young archaeological material. The fossil cementum was more variable and less dense than the fossil dentine in our sample set. A significant difference between dentine and cementum was found for the only tooth (95072) where both tissues were measured. Since the youngest true fossil sample studied thus far is about 40 ka, the increase in density of the cementum over modern values has been shown to occur within the time interval between 0.2 and 40 ka, and for dentine the increase has clearly occurred within the first 100 ka or so, but more likely within the first 50 ka (see sample 95072, Table 2).

Table 2 shows that beta doses to enamel based on One-Group theory are significantly reduced as a function of density of the dental tissues involved. ROSY ESR ages calculated using fossil cementum and dentine densities of 2.8 g cm<sup>-3</sup> were as much as 10% higher than those

calculated using modern densities of 2.1-2.2 g cm<sup>-3</sup>, whenever the cementum and enamel thicknesses are about 0.5 mm and the dominant source of dose in the sediment is potassium (because of its larger contribution of beta radiation relative to gamma). If the sediment dose arises mainly from U or Th, the age difference was lower, in the range of only 2- 5%. The density dependence on ESR age was negligible ( $\leq 2\%$ ) whenever the thicknesses of these same tissues approach 1-2 mm, and also when U uptake into dentine, cementum or enamel has occurred. Age calculations assume that density changes have occurred early in the burial history, and changes that occur later would make these effects less severe. Thus these results provide a worst-case scenario for the effects of density change on ESR ages.

### Discussion

The higher densities of cementum and dentine in fossil material do not appear to be related to rapid post-mortem dehydration because the densities of modern and 100-200 year old archaeological samples are similar to those of the modern human samples studied by Rowles (1967). However, dehydration on a longer timescale does seem to be involved in density increases. Using the values in Fig. 2, we can compute the increased density of these tissues with complete dehydration or total loss of the organic fraction, making the assumption that a commensurate volume reduction also occurs. Cementum and dentine densities would increase to 2.51 and 2.62 g cm<sup>-3</sup> respectively after dehydration, but only increase to 2.26 and 2.33 g cm<sup>-3</sup> respectively for total loss of organics. Complete dehydration could explain the increases to the fossil cementum values in Table 1, but this could not explain the increase in dentine values, which require loss of water and organic components to reach values as high as 2.89. Significant loss of organics in dentine and bone are known to occur within the first 40 ka of burial, based on yields of collagen from dentine and bone in routine radiocarbon dating. Therefore it is logical to assume that both dehydration and loss of organics are active processes during burial, and that both contribute to the increase in density of cementum and dentine.



**Figure 2.**  
Comparison of the relative proportions of mineral, organic and water components in human teeth. a) by weight %, b) by volume %.. (E) - enamel, (D) - dentine, (C) - cementum and (B) - bone, after Trautz (1967)

Before the ROSY ESR age calculation program was available, we applied fossil cementum density results to ESR dating of horse teeth from Mousterian layers at El Castillo Cave (Rink *et al.*, 1997). Here the teeth have a sediment/cementum/enamel/dentine irradiation geometry in which the cementum layer acts primarily as an absorber of sediment beta dose, without being a significant dose source itself (the cementum is virtually uranium free). The fraction,  $F$ , of infinite matrix sediment beta dose  $D_s$  transmitted through a cementum of thickness  $t$  can be approximated by:

$$F = [D_s \{ \exp(-\mu_R t p) \}] * 0.5 \quad (2)$$

where  $\mu_R$  is the linear absorption coefficient (per micrometer) in aluminium and  $p$  is the ratio of the density of aluminium to the cementum density.

We used equation 2 to calculate the fraction of infinite matrix beta dose reaching the enamel. The ages were calculated using the software program DATA (mentioned above), but we reduced the input concentration values for U, Th and K in the sediment as follows: input value =  $F * \text{measured U, Th or K concentration}$ . Without correction for the intervening cementum absorber, ESR ages were about 10% too low, since the sediment beta dose rates were erroneously high.

Experimental studies which used powdered tooth enamel as a detector of beta attenuation are in close agreement with One-Group Theory calculations of beta attenuation in solid enamel. (Rink *et al.*, submitted) Thus, we expect that future use of the density values will mainly be for applications of One-Group theory to beta attenuation in ESR dating.

### Conclusions

Density values for fossilized dental tissues are important to One-Group theory-based beta attenuation calculations for teeth with thin (ca. 0.5 mm) dental tissues. Use of values for modern teeth of  $2.1\text{--}2.2 \text{ g cm}^{-3}$  for cementum and dentine could lead to ESR age underestimates in the range of 2-10% when the dominant source of beta dose is the sediment. The mean values of  $2.54 \pm 0.22$  for fossil cementum and  $2.82 \pm 0.07 \text{ g cm}^{-3}$  for fossil dentine represent the best values available for use in One-Group Theory beta dose attenuation calculations for samples in the age range of  $>40 \text{ ka}$ . A combination of dehydration and loss of organics within the first 40 ka of burial leads to increases in density of dentine and cementum, but no changes in enamel density are found for the first 450 ka of burial for the sample set studied.

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Table 1. Densities of Modern and Fossil Dental Tissues

Tooth Number	Site	Taxa	Age (ka)	Density <sup>6</sup> Cementum (g cm <sup>-3</sup> )	Density <sup>6</sup> Dentine (g cm <sup>-3</sup> )	Density <sup>6</sup> Enamel (g cm <sup>-3</sup> )
92050	Tobermory, Canada	Horse	<0.2	2.14 ± .01 (n=2)		2.94 ± .04 (n=4)
92315	Kings Lynn, UK	Cow	Modern	2.12 ± .07 (n=3)		
94810	Williamstown, Canada	Cow	<0.2		2.22 ± .02 (n=4)	
Mean <sup>7</sup>	Young or Modern			2.13 ± .01		
94141	Staroscle, Crimea	Wild Ass	40-45 <sup>1</sup>	2.79 ± .08 (n=4)		3.02 ± .17 (n=2)
95072	El Pendo, Spain	Cow	<50 <sup>2</sup>	2.39 ± .02 (n=4)	2.89 ± .03 (n=4)	
2060	Karain, Turkey	Cow	100 - 120 <sup>3</sup>		2.76 ± .04 (n=4)	3.02 ± .05 (n=4)
91182	Krapina, Croatia	Rhinoceros	120 - 130 <sup>4</sup>		2.80 ± .07 (n=4)	2.96 ± .08 (n=4)
989	West Runton, UK	Elephant	380 - 540 <sup>5</sup>	2.44 ± .05 (n=4)		
Mean <sup>7</sup>	Fossil			2.54 ± .22	2.82 ± .07	3.00 ± .03

## Footnotes:

1. Monigal et al. (1998)
2. Preliminary ESR age, Volterra (in prep).
3. Rink et al., 1994
4. Rink et al., 1995
5. Rink et al., 1996b
6. The reported density value for a given tooth number is the average value and standard deviation (precision) of n repeated measurements.
7. The mean value is the average and standard deviation of the density measurements for the different teeth in the column directly above the quoted mean.

Table 2. One-Group Theory-based ESR age calculations for a hypothetical tooth enamel

Cementum			Dentine			Enamel		Beta Dose (EU) ( $\mu\text{Gy a}^{-1}$ )	EU ESR Age (ka)	Beta Dose (LU) ( $\mu\text{Gy a}^{-1}$ )	LU ESR Age (ka)	EU Age % Difference Low vs. High $\rho$
U (ppm)	$\rho$ ( $\text{g cm}^{-3}$ )	T (mm)	U (ppm)	$\rho$ ( $\text{g cm}^{-3}$ )	T (mm)	U (ppm)	T (mm)					
0	2.1	0.5	0	2.2	0.5	0	0.5	236	124	236	*	10
0	2.8	0.5	0	2.8	0.5	0	0.5	167	136	167		
0	2.1	0.5	0	2.2	0.5	0	0.5	46	163	46	*	2
0	2.8	0.5	0	2.8	0.5	0	0.5	33	166	33		
0	2.1	0.5	0	2.2	0.5	0	0.5	124	144	124	*	5
0	2.8	0.5	0	2.8	0.5	0	0.5	95	151	95		
0	2.1	1.0	0	2.2	1.0	0	1.0	34	166	34	*	2
0	2.8	1.0	0	2.8	1.0	0	1.0	21	170	21		
0	2.1	2.0	0	2.2	2.0	0	1.0	7	174	7	*	1
0	2.8	2.0	0	2.8	2.0	0	1.0	3	175	3		
1	2.1	1.0	1	2.2	1.0	0	1.0	60	159	46	163	2
1	2.8	1.0	1	2.8	1.0	0	1.0	49	162	33	166	
5	2.1	1.0	5	2.2	1.0	0	1.0	160	137	92	152	2
5	2.8	1.0	5	2.8	1.0	0	1.0	157	138	83	153	
20	2.1	1.0	20	2.2	1.0	20	1.0	1242	21	652	37	0
20	2.8	1.0	20	2.8	1.0	20	1.0	1262	21	656	36	

**Footnotes:**Row 1: Sediment radioactivity: U=0.5 ppm, Th = 2.01 ppm, K=1.65 wt. % (gamma dose = 568  $\mu\text{Gy a}^{-1}$ )Row 2: Sediment radioactivity: U=0 ppm, Th = 10.9 ppm, K = 0 wt. % (gamma dose = 568  $\mu\text{Gy a}^{-1}$ )All other rows: Sediment radioactivity: U= 5 ppm, Th = 0 ppm, K = 0 wt. % (gamma dose = 568  $\mu\text{Gy a}^{-1}$ )For all the calculations above: equivalent dose = 100 Gy, cosmic dose = 0  $\mu\text{Gy a}^{-1}$ , moisture in dentine = 5%, moisture in sediment = 0%, alpha efficiency = 0.15,  $^{234}\text{U}/^{238}\text{U} = 1.4$ , density of sediment =  $2.0 \text{ g cm}^{-3}$ , radon loss = 0%, enamel stripped from cementum side and from dentine side of enamel = 0.05 mm.

\* LU ages omitted because they are the same as EU for U concentrations of 0.



Reviewer

**Gunther A. Wagner**

**Comments**

This work deals with the influence of the sample's density on the beta microdosimetry in teeth. The fact that the density of the cementum and the dentine increases within the first 40 ka leads to an age underestimate if not taken into account. Although it is shown that the ultimate effect on the ESR age is in the worst case around 10% - which seems relatively little in view of errors introduced by the model assumptions on early or late uranium uptake - it is good to see a quantitative assessment of this effect.