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ESR analyses for teeth from the open-air site at Attirampakkam, India: clues to complex U uptake and paleoenvironmental change☆

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Abstract

In open-air sites, diagenetic alteration makes teeth difficult to analyze with electron spin resonance (ESR). Despite strong diagenetic alteration, three ungulate teeth from Pleistocene fluvial sediment in the open-air Paleolithic site at Attirampakkam, Tamil Nadu, India, were analyzed using standard and isochron ESR. Diagenetic alteration features in two teeth indicated rapid submergence in quiet saline to hypersaline water, following a short subaerial exposure, while the third remained constantly buried under reducing conditions. Geochemical signatures and ESR data all indicate that the teeth experienced at least three independent U uptake events during diagenesis, including two that occurred long after burial.

Keywords: ESR analyses; Mammalian tooth enamel; U uptake; Tooth diagenesis

1. Introduction

Modelling U uptake in electron spin resonance (ESR) dating continues to cause age calculation problems when the teeth have absorbed substantial U (Falguères, 2003). Teeth that show U leaching require more complicated U mobility models (Hoffmann and Mangini, 2003). Isochron analysis, however, can help unravel complex U uptake histories in uraniferous teeth (e.g., Blackwell et al., 2001).

Most ESR studies have used teeth from caves (Falguères, 2003), because in open-air sites, such as Attirampakkam, rapid, pervasive, fossil diagenesis and weathering produce teeth that are harder to prepare and analyze using ESR. Enamel diagenetic alteration reduces ESR peak heights, but not age accuracy (Skinner et al., 2000). Contamination by Fe, Mn and Ti, which interfere with the hydroxyapatite (HAP) signal, however, can cause inaccuracy in peak height measurements. Badly altered teeth can show all these problems and suffer multiple U uptake events.

At Attirampakkam, in the Tiruvallur district, South India, an open-air site yielded Paleolithic artifacts from deeply buried Pleistocene fluvial sediment. Although known for a century, Attirampakkam's Acheulian–late Middle Paleolithic sequence has never been successfully dated (Pappu, 2001a). As one of India's few stratified Paleolithic sites, its dates are critical to understanding Indian Paleolithic cultures. In an ephemeral gully near the Kortallayar River, Attirampakkam (Fig. 1) sites 47 km inland from the modern shoreline (13°13′50″N 79°53′20″E, 37.5 m asl; Pappu, 2001a, b). Deposited on a floodplain, possibly in a pond or crevasse splay, Layer 6

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Table 1
Attirampakkam teeth in the study

ESR sample	Test pit	Layer	Quad	Depth (m)	Species	Tooth type
FT28	6	5	C2	2.80	*Equus*	Molar
CT63	3	Eroding from 6	–	Surface	Bovid, *Bulabus?*	Molar
FT20	3	6	B2	5.90	Caprinae or Boselephini	Molar

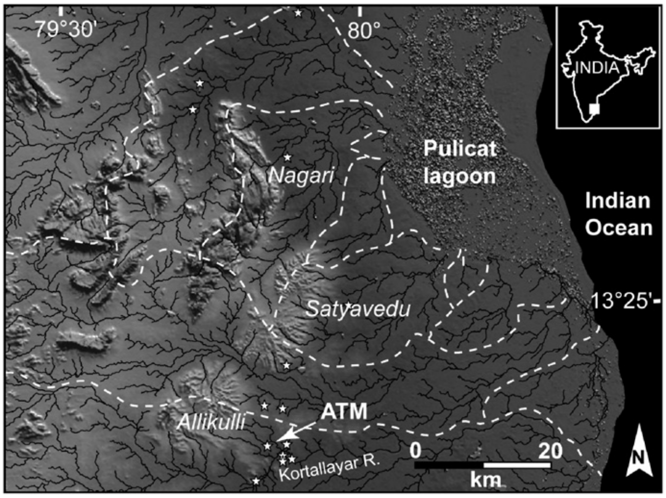

Fig. 1. Attirampakkam, South India Attirampakkam (ATM) is located on a tributary of the Kortallayar River, in Tiruvallur district, Tamil Nadu, South India.

contains laminated silty clay with thin carbonate lenses at the base, disconformtably overlain by thick ferruginous gravels (Layer 5). Layer 6 yielded Acheulian industries, and Layer 5, early Middle Paleolithic. Pappu's excavations (1999–2004) yielded three fossil teeth from Layers 5 and 6 (Table 1; Pappu et al., 2003, 2004). Despite their conspicuous diagenetic alteration, all three teeth were analyzed by standard and isochron ESR to detail their U uptake histories and to provide the first dates for the site. By studying teeth with complex U uptake histories, better U uptake modelling procedures can be developed.

2. Analytical method

The teeth (Table 1, Fig. 2) were prepared using standard and isochron ESR methods (Blackwell, 1989). Four enamel subsamples from FT28 were split into unaltered (W) and diagenetically altered (B) enamel subsamples to assess how diagenesis affected enamel geochemistry, ESR signals and age variation. After adding γ doses ranging from 0 to 2560 Gy, at 120–140 Gy/s, all aliquots were annealed at 90 °C for 3.0 days to remove unstable interference (Skinner et al., 2000). All aliquots were measured at 20 °C in a JEOL RE1X spectrometer at 2 mW power, under a 100 kHz field modulation of 1.0 mT using a 0.3 s time constant. Spectra were scanned over 10 mT centered at 360 mT with an 8.0 min sweep time. Due to the sedimentary inhomogeneity at Attirampakkam, external dose rates, $D_{\text{ext}}\ (t)$, were determined by volumetric averaging of neutron activation analyses (NAA) for several associated sediment samples (Table 2; Blackwell and Blickstein, 2000). Standard peak heights for the hydroxyapatite peak at $g = 2.0018$ were measured from derivative spectra without deconvolution (Skinner et al., 2001). Accumulated doses (A_Σ) and their errors were calculated by fitting data to a saturating exponential with a $1/I^2$ weighting. All ages, dose rates and their errors were calculated with Rosy v. 1.4, using a cosmic dose rate of $107 \pm 10\,\mu$Gy/y (Brennan et al., 1997).

3. Results and discussion

During subaerial weathering, "green bone" fractures cracked CT63 and FT20, but the pieces had separated little or not all (0–5 mm) before sediment filled the cracks and secondary carbonate cemented the pieces together in nearly perfect orientation (Fig. 2). Such fractures indicate that the teeth were subaerially exposed for a few months shortly after death (Behrensmeyer, 1978). Dating these teeth, therefore, dates the water level rise that submerged them. With low Th, K and relatively low U concentrations (Table 2), the associated secondary carbonate cements occur most commonly in coastal and fluvial–deltaic environments at the saline–fresh groundwater interface. The carbonate cement and sediment inside the teeth imply that the water levels rose rapidly, but with little or no turbulence, submerging these teeth before they had been moved or broken apart. After their submergence, possibly in a still pond or swamp, sandy, silty carbonate sediment seeped into the teeth and the secondary cement crystals grew slowly without displacing the pieces. All CT63 and FT20 ESR spectra showed no measurable aragonite or calcite signals, indicating that all secondary carbonate sediment had been removed during enamel preparation.

During weathering, FT28 experienced external remineralization, mainly Fe and Mn oxide staining, due to reducing water conditions under low O fugacity and an acidic pH. Since this indicates that this tooth was rapidly submerged soon after death and then continuously buried (Blackwell et al., 2000), dating FT28 dates when the watertable reached Layer 5. When scanned, all FT28 subsamples had normal HAP spectra with no Fe or Mn interference, indicating that the enamel subsamples had not been contaminated by the ferricalcrete coating the tooth.

The only preparation problem arose in obtaining pure dentine subsamples for NAA. Because pure dentine was scarce, most dentine subsamples contained some sediment contamination, but visually pure dentine subsamples were also collected where

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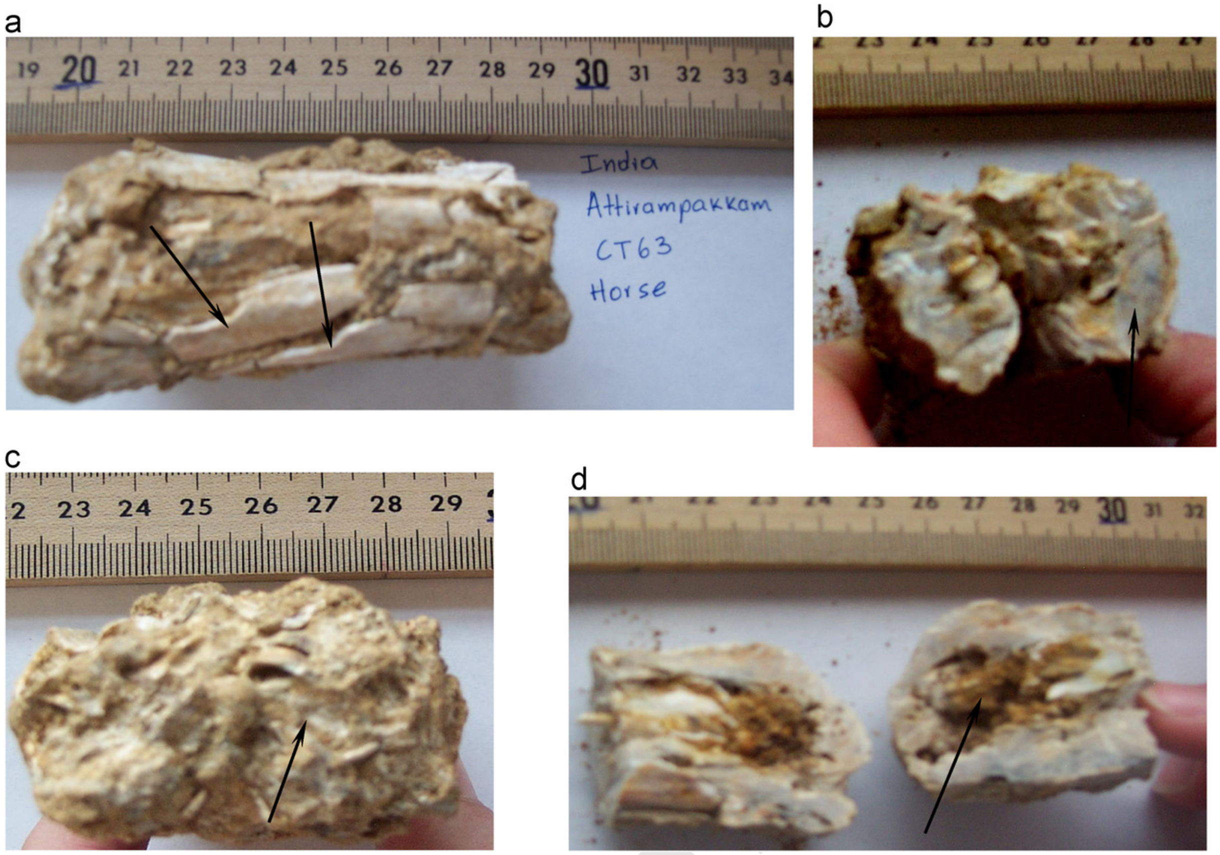

Fig. 2. Diagenetic features in CT63. CT63 shows diagenetic features typical of subaerial exposure followed by rapid burial: (a) green bones fractures occur at the arrows. Many others were noted. These only form if the tooth is exposed for several months immediately after death. The fractures show minimal separation, suggesting that the tooth was not moved at all after fracturing before cementation. (b) Cutting parallel to the occlusal surface revealed sediment cemented by a lightly colored secondary carbonate (at the arrow and other points) that binds both the whole tooth together, again showing that tooth was not moved between fracturing and cementation. This cement commonly forms in the zone where saline water meets fresh groundwater. (c) On the occlusal surface, the green bone fractures penetrate both enamel and dentine (at the arrow and other points), suggesting prolonged exposure (cf. Behrensmeyer, 1978). (d) Longitudinal cutting showed not only the secondary cement, but also terra rosa (arrow) in the pulp cavity. This might have entered the tooth before or after fracturing but would have been easily eroded if the tooth had not been rapidly inundated by quiet water after the fracturing. Together, the evidence indicates exposure for at least a year, followed by rapid submergence by deep quiet water in an environment close to the edge of the freshwater lens in a fluvial environment with occasional saline water incursions.

the dentine showed minimal secondary carbonate under UV magnification.

The high enamel U concentrations (Table 3)indicate that the teeth spent some years immersed in saline to hypersaline water, as typical in subtropical saline lakes (Blackwell et al., 2000). Usually, the dentine/enamel U concentration ratio averages 10:1 (Grün and Taylor, 1996). The CT63 and FT20 enamels had higher U concentrations, because most dentine subsamples contained some secondary carbonate sediment. Pure dentine averages 0.02 wt% K (Blackwell et al., 2002), but the Attirampakkam floodplain sediment contains substantial K thanks to the marine Avadi shale. Since the visually pure dentine subsamples contained no measurable Th or K, they probably had insignificant carbonate contamination. In CT63's mixed sediment and dentine subsamples (Table 2), U and Th were both fairly uniform. Their highly variable K (0.01–1.14 ppm), however, suggests variable amounts of saline carbonate contamination, possibly derived from the adjacent and underlying Cretaceous Avadi shale or its saline groundwater.

Unlike CT63 and FT20, FT28's dentine contained more U than its enamel (Table 3), suggesting different U uptake conditions in Layers 5 and 6. Its U concentration variation suggests that FT28 suffered at least two distinct secondary U uptake events (Blackwell et al., 2000). The outer, diagenetically altered, B enamels absorbed more U than their unaltered W counterparts (Fig. 3). During the earlier secondary U uptake event, the altered subsamples, especially FT28en3B, absorbed significant U, which caused dissimilar accumulated doses, A_Σ, but similar standard ages, for FT28en3W and FT28en3B. Later in FT28's history, some altered B enamels again absorbed more U, especially FT28en1 and FT28en4. This caused their current U concentrations to differ more, but their A_Σ's to differ little, compared to the subsamples affected by only the earlier secondary event. For the FT28en1 and FT28en4 pairs, however, their standard ESR ages differ significantly, since the subsamples had similar U concentrations throughout most of their history. The increased U concentrations in the B subsamples resulted in apparently lower ages, because having been added

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Table 2
Radioactivity in the Attirampakkam sediment

Subsample type (number)	Pit unit		Concentrations			Dose rates[a]	
			U (ppm)	Th (ppm)	K (wt%)	$D_{ext,.\beta}(t)$[b] (mGy/y)	$D_{ext,.\gamma}(t)$[c] (mGy/y)
2004.03, bulk	T7B		1.84	6.20	0.16	0.078	0.384
carbonate (1)	3/4	±	0.02	1.10	0.01	0.009	0.048
2004.06, clay	T7B		2.63	13.53	0.50	0.156	0.756
+ ferricrete (1)	4	±	0.02	0.84	0.01	0.017	0.065
2004.05 bulk	T6		4.52	16.42	0.40	0.196	0.986
ferricrete (1)	5	±	0.02	1.03	0.01	0.021	0.085
FT28 den + sed	T6		48.53	0.98	0.06	1.033	3.761
mean (6)	5	±	5.98	0.85	0.01	0.162	0.544
2004.04 clay,	T3		2.89	18.84	1.34	0.268	1.099
laminated (1)	6	±	0.02	1.21	0.04	0.029	0.086
CT63sed + den	T3		10.52	4.07	0.20	0.273	0.981
attached (4)	6	±	9.56	4.22	0.16	0.217	0.751
CT63	T3		6.88	1.91	0.20	0.184	0.626
“dentine” (8)	6	±	1.11	0.37	0.35	0.050	0.114
FT20sed,	T3		3.33	6.06	0.25	0.118	0.508
attached (1)	6	±	0.02	0.37	0.04	0.013	0.042
FT20 den + sed	T3		22.95	3.83	0.19	0.676	2.497
(4)	6	±	10.09	0.16	0.01	0.224	0.797
Detection		~	0.01	0.20	0.001		
limits[d]		–	0.02	0.40	0.002		

[a] Assuming water concentration $W_{sed} = 25 \pm 5$ wt%.

[b] Assuming enamel density $\rho_{en} = 2.95 \pm 0.02\,g/cm^3$, dentine density $\rho_{den} = 2.85 \pm 0.02\,g/cm^3$, cementum density $\rho_{cem} = 2.85 \pm 0.02\,g/cm^3$, sediment density $\rho_{sed} = 2.66 \pm 0.05\,g/cm^3$, enamel water concentration $W_{en} = 2 \pm 2$ wt%, enamel thickness $\phi_{en} = 1.000 \pm 0.100$ mm, enamel removed ϕ_{-in}, $\phi_{-out} = 0.020 \pm 0.005$ mm.

[c] Assuming cosmic dose rate $D_{cos}(t) = 0.000 \pm 0.000$ mGy/y.

[d] Detection limits depend on sample mass and mineralogy.

Table 3
U concentrations in the Attirampakkam teeth

Subsample (number of analyses)m		U concentrations			
		Unaltered enamel (ppm)	Altered enamel (ppm)	Inner dentine (ppm)	Inner dentine+ Sediment (ppm)
FT28 min.		25.34	28.77	51.21	39.02
FT28 max.		34.82	42.94	61.55	56.47
FT28 mean		31.17	36.24	52.84	47.02
(12)	±	5.11	6.08	4.95	5.98
CT63 min.		14.88	–	11.68	5.94
CT63 max.		21.03	–	26.76	8.66
CT63 mean		17.62	–	–	6.88
(9)	±	2.11	–	–	1.11
FT20 min.		35.56	–	–	12.07
FT20 max.		45.98	–	–	34.88
FT20 mean		40.77	–	–	22.95
(6)	±	7.37	–	–	10.09
Typical errors	±	0.02	0.02	0.02	0.02
Detection	~	0.01	0.01	0.01	0.01
limits	–	0.02	0.02	0.02	0.02
Typical water		5.5	10.10	.	
concentrations	±	2.2	2.2		

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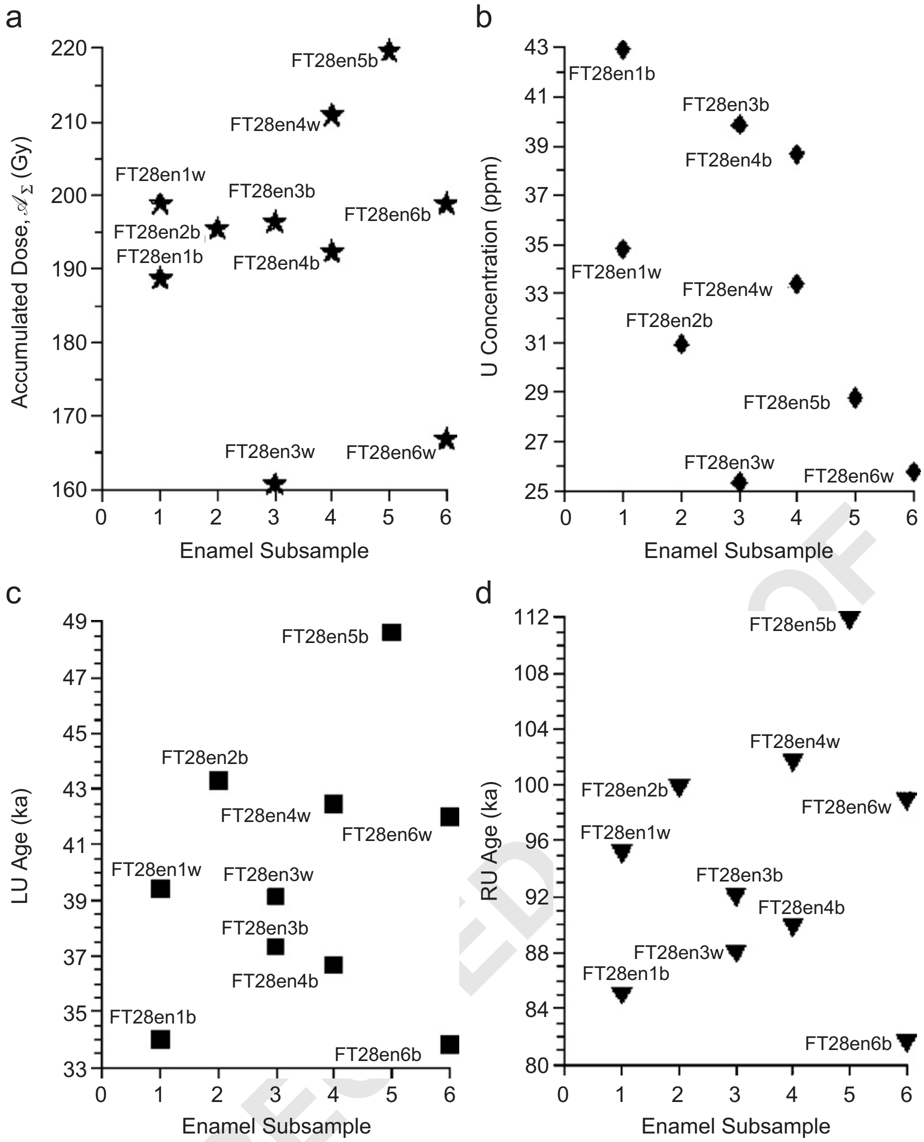

Fig. 3. Effects from U uptake events during diagenesis on the FT28 enamel. Since all subsamples must be of the same age, secondary U uptake by the enamel or dentine during diagenesis must have caused the differences seen here: (a) accumulated doses, A_Σ: In FT28en1 and FT28en4, A_Σ for the diagenetically unaltered (W) enamel exceeded A_Σ for the altered (B) by ~ 10 and 18 Gy, respectively. The altered FT28en3B subsample has the larger A_Σ by ~ 38 Gy, while FT28en6B is ~ 30 Gy larger than FT28en6W. (b) U concentrations: Diagenetically altered (B) enamels contained more U than unaltered (W) enamels. The greatest difference between the B and W subsamples occurs in FT28en3 (~ 15 ppm), while FT28en1 differs by about 8 ppm, and FT28en4, by 4 ppm. (c) LU ages: The smallest difference in occurred between FT28en3B and FT28en3W (~ 2 ky), with FT28en3W apparently older. For FT28en1 and FT28en4, the B and "older" W ages differed by ~ 3.2 ky. (d) RU ages: The smallest difference again occurred in FT28en3, but FT28en3B is apparently older. For FT28en1 and FT28en4, the age differences have increased, but the W's still appear "older". This hints that FT28en3B and FT28en6B absorbed extra U early in their histories. That early extra U affected their A_Σ's throughout most of their history, making their A_Σ's substantially higher than their W counterparts, but giving them similar ages. In FT28en1B and FT28en4B, the secondary U uptake occurred late in their histories. That late extra U only affected their A_Σ's for the later part of their histories, making their A_Σ values much more similar, but making their ages different.

late, the U did not contribute to A_Σ throughout the tooth's history.

For CT63, isochron analysis yielded an LU age of 44 ± 11 ka. Its $D_{\text{ext}}(t)$ was somewhat higher than, but agreed within the errors with, that from the volumetric geochemical analyses (Fig. 4a), suggesting that any major U remobilization occurred late in its history. CT63en1's coupled ESR-^{230}Th/^{234}U, 53 ± 11 ka, corroborates this. Two subsamples, however, suggest that some recent minor secondary diffusional U remobilizaton affected CT63 late in its history (cf. Blackwell et al., 2001). This may have occurred if the tooth was reworked or when modern erosion exposed it. A preliminary coupled ESR-^{230}Th/^{234}Uage for CT63en1, 53 ± 7 ka, corroborates this.

For the unaltered FT28 enamels, the LU isochron gave 48 ± 12 ka (Fig. 4b), which agreed well with the standard LU age

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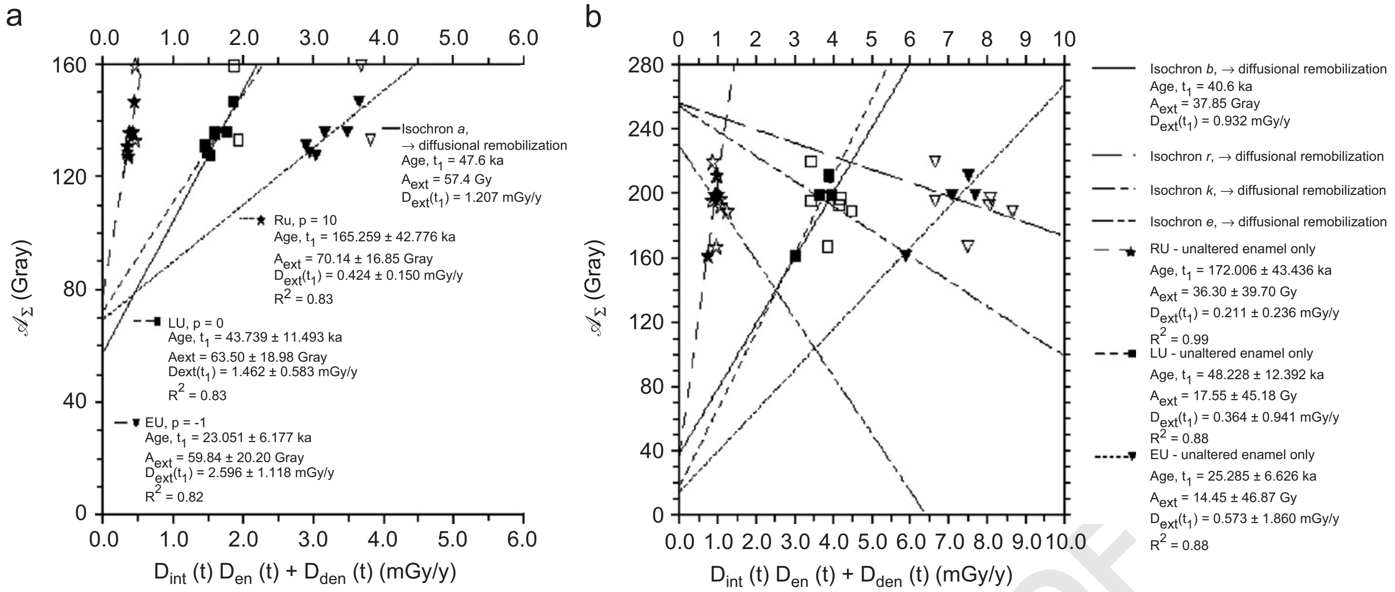

Fig. 4. ESR isochron analyses for CT63 and FT28 at Attirampakkam, India. Both isochrons show evidence for two or more U uptake events (cf. Blackwell et al., 2000, 2002). For the LU isochrons: (a) with six of its eight subsamples, CT63's isochron shows good correlation, $R^2 = 83\%$. The isochron's $D_{\text{ext}}(t) = 1462 \pm 523\,\mu\text{Gy/y}$ is slightly higher than that measured in the associated sediment ($1206 \pm 96\,\mu\text{Gy/y}$). The isochron's age, 43.4 ± 11.4 ka, also is just slightly lower than the standard age, 47.6 ± 1.4 ka. Isochron ***a*** shows the LU parameters for the standard analyses, which do not differ significantly from the isochron results. Two subsamples (open points), however, hint that the tooth has suffered some secondary diffusional remobilization late in its history. (b) In FT28, the unaltered W enamels (closed points) give an isochron with a positive slope and an age of 48 ± 12 ka. The $D_{\text{ext}}(t)$, $364\,\mu\text{Gy/y}$, is slightly lower than, but not significantly different from, the current $D_{\text{ext}}(t)$ (isochron ***b***), hinting at some late secondary diffusional U remobilization, as does the negative slope for the altered B enamel isochrons (open points). Both secondary events included some secondary uptake and some leaching depending on the subsample. For both, different paleoenvironmental conditions after a reworking event may have caused the secondary U remobilization.

Table 4
Weighted mean ages for teeth in the study

Sample (number)		Accumulated dose, A_Σ (Gy)	Standard ESR ages[a] EU[b] (ka)	LU[b] (ka)	RU[b,c] (ka)
FT28		193.6	23.6	40.6	95.3
unaltered	±	2.8	1.0	1.6	3.6
(4)		1.4%	4.3%	4.0%	3.7%
FT28		198.2	23.1	40.0	95.2
altered	±	2.3	0.9	1.5	3.2
(6)		1.1%	4.0%	3.7%	3.4%
CT63		140.8	30.3	47.6	83.4
(8)	±	1.6	1.0	1.4	2.31
		1.1%	3.3%	3.0%	2.7%
FT20		111.0	14.7	25.7	64.3
(2)	±	1.8	1.0	1.7	3.5
		1.6%	7.0%	6.5%	5.4%

[a] Assuming α efficiency $k_\alpha = 0.15 \pm 0.02$, activity ratio $(^{234}\text{U}/^{238}\text{U})_0 = 1.20 \pm 0.20$, water concentration $W_{\text{en}} = 2 \pm 2$ wt%, water concentration $W_{\text{den}} = 5 \pm 2$ wt%, enamel density $\rho_{\text{en}} = 2.95 \pm 0.02\,\text{g/cm}^3$, dentine density $\rho_{\text{den}} = 2.85 \pm 0.02\,\text{g/cm}^3$, radon loss from the tooth $\text{Rn}_{\text{tooth}} = 0 \pm 0\%$, sediment density $\rho_{\text{sed}} = 2.65 \pm 0.02\,\text{g/cm}^3$.

[b] Water concentration $W_{\text{sed}} = 25 \pm 5$ wt%, cosmic dose rate $D_{\text{cos}}(t) = 107.4 \pm 10.0\,\mu\text{Gy/y}$.

[c] U uptake parameter, $p = 10$.

(Table 4). Although slightly lower, the isochron's $D_{\text{ext}}(t)$ did not differ significantly from the modern $D_{\text{ext}}(t)$. The difference, however, may hint at some late secondary diffusional U remobilization (cf. Blackwell et al., 2002), which affected FT28en1W, FT28en4W, and FT28en6W, more than FT28en3W. The diagenetically altered enamels, however, gave isochrons with negative slopes, indicating that, late in their history, they experienced diffusional "hot atom" U uptake and remobilization, which affected FT28en1B, FT28en4B and FT28en6B more than the others. In "hot atom" remobilization, subsamples which initially contained more U are affected more during subsequent events, because radioactive decay around the uraniferous sites produces more porosity, especially from α tracks, through which water can reach those sites (Blackwell et al., 2001, 2002).

The very high U concentrations in FT20 compared to those in CT63 hint that FT20 may have also absorbed about half its U late in its history. Because CT63 sat exposed on the surface, it likely did not retain the U from the most recent U uptake (RU) event, because weathering would have leached any adsorbed U easily. CT63en1 experienced uptake with $p = -0.65 \pm 0.19$ (between EU and LU), although the dentine more likely had more linear uptake, whereas FT28 and FT20 likely have U uptake models between $p = 0$ (LU) and $p = 10$ (RU). More coupled ESR-$^{230}\text{Th}/^{234}\text{U}$ dates are needed to confirm these uptake models, but the multiple U uptake events could complicate those coupled age calculations. Therefore, the tooth ages reported here likely underestimate those for the stratigraphic layers and their artifacts. With only three teeth dated from the two layers, it remains impossible to test for reworking, which some post-depositional bioturbation features hint may have affected the teeth.

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4. Conclusions

The diagenetic alteration features in FT20 and CT63 indicated a short exposure followed by rapid burial, likely in still, saline to hypersaline water, while those in FT28 indicated constant burial in a reducing environment, as typical on floodplains. Meanwhile, the geochemical signatures and ESR signals recorded by the teeth all indicate that CT63 and FT28, and presumably FT20 also, experienced multiple U uptake events that produced complex patterns of U concentrations, accumulated doses and apparent ages across the teeth. Isochron analyses coupled with such detailed ESR analyses on differently altered tooth enamel subsamples can illuminate a complex U uptake history. Although poor preservation may be the rule for fossil teeth in open-air sites, it does not preclude ESR analyses that can provide valuable geochronological and geochemical insight into the diagenetic processes that affect such sites.

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