Optically stimulated luminescence dating of Ocean Drilling Program Core 658B: Complications arising from authigenic uranium uptake and lateral sediment movement.

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Abstract

Ocean Drilling Program Site 658 lies under the North African summer dust plume, and ought to be an ideal target for optically stimulated luminescence (OSL) dating, since the main clastic input is far-travelled Saharan dust. However, OSL ages for coarse silt-sized quartz (40-63 μm) are systematically lower than independent age estimates when dose rates are calculated using a model which assumes detrital ^{238}U, ^{232}Th and ^{40}K and excess ^{230}Th and ^{231}Pa. Ages which are in good agreement with independent age control are obtained from the coarse silt samples when a correction for authigenic uranium uptake is incorporated into the dose rate model. Authigenic uranium uptake occurs under reducing conditions, which are common at the sediment-water interface, and some degree of authigenic uranium correction may be required for most marine sediments. Using this revised dose rate model, ages produced using fine silt-sized quartz (4-11 μm) are up to 100% older than both independent and coarse silt ages. In addition, the fine silt ages show a consistent pattern of age decrease with depth over 1.5 m of core. ^{230}Th data from Site 658 indicate that this site receives 3 times more sediment laterally than vertically. It is concluded that the fine silt at Site 658 contains a substantial reworked component, making it unsuitable for dating. Conversely the coarse silt fraction, which settles through water at ~40 times the rate of fine silt, appears to be derived from dust input over the site at the time of deposition. Since prominent nepheloid (cloudy) layers occur in

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various deep ocean basins, and the material suspended in these layers often consists of reworked fine silt-sized sediments, coarser material should be dated where possible.

Keywords: Optically stimulated luminescence; Geochronology; Marine sediments; North Africa.

1. Introduction

Marine sediments are one of the most widely exploited archives of palaeoenvironmental information. At present, most chronologies are constructed by tuning marine proxies for global ice volume ($\delta^{18}O$) to the well understood variations in the Earth's orbit (Lisiecki and Raymo, 2005), by the identification of event horizons (e.g. Sikes et al., 2000) and/or by radiocarbon dating. However, dating deep-sea sediments remains problematic. Chronologies based on $\delta^{18}O$ records are dependent upon the accuracy of the climate change model assumed and necessarily obscure information regarding phasing of the events observed. Event horizons are only useful where their occurrence is reasonably frequent in the time period of interest. In addition, both techniques require approximately continuous sedimentation, and the absence of sediment reworking. Whilst a tremendously powerful geochronological tool, Accelerator Mass Spectrometry (AMS) radiocarbon dating is limited to the last 50-60,000 years and is subject to uncertainties due to variation in the atmospheric ^{14}C concentrations and the marine carbon reservoir. The latter effect may introduce a substantial and temporally variable systematic error to a chronology (Sikes et al., 2000). In addition, chronologies can only be constructed using radiocarbon or the $\delta^{18}O$ record where organic carbonates are preserved, which is not the case in areas with high clastic sedimentation rates (Sugisaki et al., 2010), or at sites below the carbonate compensation depth. These limitations to well established dating techniques have led several authors (Berger, 2006; Jakobsson et al., 2003; Stokes et al., 2003) to advocate the use of optically stimulated luminescence (OSL) methods to provide age control for deep-sea sediments.

Although marine sediments were first dated using thermoluminescence techniques in 1979 (Wintle and Huntley, 1979) and using OSL techniques in 2003 (Jakobsson et al., 2003; Stokes et al., 2003), there are relatively few published luminescence chronologies for open ocean sediments. This absence is possibly explained by the complex dose rate measurements and calculations required, due to the presence of uranium-series disequilibrium in many deep sea sediments (Wintle and Huntley, 1979). For many terrestrial sediments it is valid to assume that the ^{238}U, ^{235}U and ^{232}Th decay series are in secular equilibrium. Consequently, dose rates due to these decay series are effectively invariant over Quaternary timescales. This remains true for ^{232}Th in marine sediments since thorium is highly insoluble and is incorporated into marine sediments, in equilibrium with its daughters, within the detrital component. Conversely, the geochemistry of both uranium decay series makes it likely that they will be in disequilibrium in late Quaternary ocean sediments. In the oxidising conditions found in most seawater, uranium is highly soluble. However, long-lived isotopes in both uranium decay series (^{230}Th in the ^{238}U series and ^{231}Pa in the ^{235}U series) are insoluble and are removed from the water column by sorbtion to the surface of settling particles (Henderson and Anderson, 2003) meaning that they are initially found in excess (relative to isotopes earlier in the relevant decay series) in many marine sediments. These isotopes have half-lives which are similar to the timescales over which OSL dating is applicable (75 and 32.8 ka for ^{230}Th and ^{231}Pa respectively) causing the dose rate due to these isotopes and their decay products to evolve as the sediment ages. Consequently, OSL dating of marine sediments is complicated by the need to demonstrate the absence of excess activity in the uranium decay series (Jakobsson et al., 2003; Sugisaki et al., 2010; Sugisaki et al., 2012) or to quantify excess activity and incorporate the resulting time-dependant dose rate changes into age calculations (Stokes et al., 2003; Wintle and Huntley, 1979). This study compares AMS ^{14}C and quartz OSL chronologies produced for a core from Ocean Drilling Program (ODP) Site 658.

2 Materials and methods

Core 658B was the second of three cores recovered from a water depth of 2,263 m off Cap Blanc, Mauritania (20°45'N, 18°35'W) during ODP Leg 108 (Ruddiman et al., 1988). Trade winds cause strong upwelling over the site, leading to high surface productivity and high biogenic particle fluxes to the seafloor. Biogenic carbonate comprises 40-60% by mass of the sediment, with the remainder being terrigenous dust (deMenocal et al., 2000). The high terrigenous dust flux is due to the site's location beneath the axis of the summer African dust plume (Figure 1). Core 658B is an ideal target for testing OSL dating in the marine realm since: (1) It has a high accumulation rate (c.18 cm/ka, deMenocal et al., 2000); (2) Terrigenous dust input should provide a substantial well-bleached quartz component; (3) Neighbouring core 658C has a well dated dust flux record covering the last 22 ka (deMenocal et al., 2000) and (4) the excess ^{230}Th ($^{230}Th_{xs}$) record for core Core 658C (Adkins et al., 2006) may be used to calculate excess activity in Core 658B.

Independent chronological data for Core 658B were obtained from two sources. Firstly, 18 samples from core depths ranging from ~2.3-10.9 m (effectively the upper ~8.5 m of core since the uppermost 2.3 m appears to replicate underlying material) were dated by AMS ^{14}C on 150-250 µm *Globigerinoides bulloides* tests. Fifteen of these AMS ^{14}C measurements yielded finite ages (Table S1). Secondly, dated stratigraphic markers in Core 658C (deMenocal et al., 2000) and the LR04 benthic δ^{18}O stack (Lisiecki and Raymo, 2005) were identified in Core 658B (Table S2). Both approaches yield a similar age-depth relationship, though the Core 658B AMS ^{14}C ages show age inversions especially below 7 m, towards the upper limit of the technique (Figure 2). There is no apparent lithological change at 7 m, and it is possible that the age inversions are due to reworking of foraminiferal tests or variable reservoir effects.

2.1 Equivalent dose determination

Samples were obtained from a 66 mm diameter split core at the ODP East Coast Repository, Lamont-Doherty Earth Observatory, USA. Since collection in 1986, the core had been refrigerated

and kept moist via damp sponges inserted at the ends of each core section, but no attempt had been made to shield the core from light. Paired samples were taken every ~25 cm through the uppermost 11 m of the core by inserting short sections of 20 mm diameter opaque tubing. Under subdued orange lighting the sediment was extruded and the outer ~8 mm was discarded to avoid core barrel smearing. The light exposed upper ~8 mm was removed and used for carbonate content and dose rate analysis. The middle ~8 mm of the sample was dispersed in deionised water and sieved at 150 μm, with the >150 fraction consisting of foraminiferal tests, and the <150 μm fraction being used for dating. The dating fraction was sequentially treated with HCl, H_2O_2 and H_2SiF_6 to remove carbonate, organic matter and feldspars respectively. The resulting mixture was separated into fine silt (4-11 μm) and coarse silt (40-63 μm where present) fractions via Stokes settling and wet sieving respectively.

All OSL measurements presented in this study were carried out using a Risø TL/OSL-DA-15 automated dating system (Bøtter-Jensen et al., 2000). Optical stimulation of single aliquots was carried out using a blue (470 ± 30 nm) light emitting diode (LED) array with a nominal power density of 18 mW/cm^2. Infra-red (IR) stimulation was carried out using an IR (870 nm) laser diode array. OSL was measured using an Electron Tubes Ltd 9235QB photomultiplier tube with 7.5 mm of Hoya U-340 filter interposed between the sample and photomultiplier. Irradiation was carried out using a 40 mCi $^{90}Sr/^{90}Y$ beta source, calibrated relative to the National Physical Laboratory, Teddington Hotspot 800 ^{60}Co γ-source (Armitage and Bailey, 2005). Single-aliquot equivalent doses (D_e) were determined using the single-aliquot regenerative-dose (SAR) method (Galbraith et al., 1999; Murray and Wintle, 2000). OSL signals were measured at 125 °C and growth curves were fitted using a saturating exponential function. No dependence of D_e upon preheating regime was found (Figure S1) and a preheating regime of 260 °C, 10 s for PH1 (the pre-heat prior to measurement of L_n or L_x) and 220 °C, 10 s for PH2 (the pre-heat prior to measurement of T_n or T_x) was adopted for subsequent measurements. Dose recovery experiments (Roberts et al., 1999; Wallinga et al., 2000) were

performed on the coarse silt fractions of samples 11B, 19A and 27A (D_e = 23, 44 and 77 Gy respectively) using this preheating regime, yielding dose recovery ratios of 0.99 ± 0.01, 0.98 ± 0.01 and 0.98 ± 0.01 respectively. When calculating D_e, aliquots were rejected where the recycling ratio (Murray and Wintle, 2000) or IR depletion ratio (Duller, 2003) differed from unity by more than two standard deviations, or where the sensitivity corrected luminescence intensity in response to a 0 Gy regeneration dose exceeded 5% of the sensitivity corrected natural luminescence intensity (Table S3).

2.2 Dose rate detremination

The environmental dose rate for samples from core 658B consists of alpha, beta and gamma components, since the overlying water completely shields the ocean floor sediments from cosmic rays. ^{238}U and ^{232}Th and bulk K concentrations were measured using ICP-MS (Table S4). Burial $^{230}Th_{xs}$ for all samples was assumed to be the mean value (36.6±9.3 Bq/kg) measured by Adkins et al. (2006) for Core 658C over the time period 2-18.5 ka. Adkins et al. (2006) determined $^{230}Th_{xs}$ using a VG Plasma Quad 2 ICP-MS, reporting <1% counting statistics errors and 0.17 Bq/kg procedural blanks. Initial $^{231}Pa_{xs}$ (3.38±0.87 Bq/kg) was calculated using the 0.093 production activity ratio of $^{231}Pa/^{230}Th$ (Henderson and Anderson, 2003). Time independent dose rates were calculated from ^{238}U and ^{232}Th and K concentrations, assuming equilibrium in both decay series, using the standard conversion factors (Adamiec and Aitken, 1998). The additional time dependant dose contribution due to $^{230}Th_{xs}$ and $^{231}Pa_{xs}$, and the final age calculation, were performed using the method outlined by Stokes et al. (2003). Both coarse and fine silt dose rates were corrected for alpha efficiency (0.04±0.02), alpha and beta attenuation and water content (Aitken, 1985). Water contents were taken from the relevant ODP initial report (Ruddiman et al., 1988). The above dose rate calculation is referred to as the "$Marine_{xs}$" dose rate model hereafter. Dose rates and ages calculated using the $Marine_{xs}$ model are presented in Table S5.

3 Results and discussion

Coarse silt OSL ages calculated using the $Marine_{xs}$ model are shown alongside the independent chronological data in Figure 2. It is clear that the coarse silt OSL ages underestimate the independent ages by a considerable amount.

Whilst uranium forms soluble species in oxidising conditions, under suboxic or anoxic conditions it is reduced to its insoluble tetravalent state (Henderson and Anderson, 2003). This situation frequently occurs at or near the sediment-water interface, resulting in the incorporation of authigenic uranium in marine sediments. Especially high authigenic uranium concentrations occur where high surface productivity leads to a high organic matter flux to the sea bed, since this material consumes oxygen during its decay (Henderson and Anderson, 2003). Consequently it might be expected that sediments from ODP site 658 will have a high authigenic uranium content. Authigenic uranium isotopes ($^{238}U_{auth}$ and $^{234}U_{auth}$) are incorporated into marine sediment at the seawater activity ratio of $^{234}U/^{238}U = 1.146$ (Robinson et al., 2004), and without any supported decay products. For dating purposes, $^{238}U_{auth}$ instantly attains equilibrium with the next two decay products (^{234}Th and ^{234}Pa) since neither is long-lived. Conversely, ^{234}U has a half-life of 245 ka and hence will not approach equilibrium with its parent (^{234}Pa) over the applicable age range of quartz OSL dating. However, since authigenic uranium is incorporated into the sediment at an activity ratio of $^{234}U/^{238}U$ of 1.146, $^{234}U_{auth}$ may be treated as being in equilibrium with $^{238}U_{auth}$ for the purposes of estimating dose rates. The slight excess activity of $^{234}U_{auth}$ over $^{238}U_{auth}$ has a negligible impact upon dose rates since the decay of ^{234}U contributes a small proportion of the dose rate due to the ^{238}U decay series (11% of alpha, 0.5% of beta and 0.1% of gamma). Since both ^{234}U and its decay product ^{230}Th are long-lived (half-lives of 245 and 75 ka respectively), the ingrowth of $^{234}U_{auth}$ decay products is negligible over the 0-50 ka timeframe covered by this study. Consequently, in this study the dose rate due to authigenic uranium may be approximated by assuming secular equilibrium from $^{238}U_{auth}$-$^{234}U_{auth}$, and no dose from the decay products of $^{234}U_{auth}$. Since most of the alpha, beta and gamma energy in the ^{238}U decay series is emitted at or below ^{234}U

(Stokes et al., 2003), the dose rate must be corrected to account for authigenic uranium uptake where it occurs.

The authigenic uranium content of a marine sediment may be calculated from the measured ^{238}U and ^{232}Th activities (or the activities calculated from the measured concentrations), since the ^{232}Th is entirely detrital, and the $^{238}U/^{232}Th$ activity ratio of crustal rocks and pelagic marine sediments is 0.8±0.2 (Anderson et al., 1989). Equation 1 was used to calculate authigenic uranium contents of Core 658B samples

$$U_{auth} = {}^{238}U_m - 0.8 * {}^{232}Th_m \quad \text{(Eq. 1)}$$

where $^{238}U_m$ and $^{232}Th_m$ are the measured activities of ^{238}U and ^{232}Th respectively (Yu et al., 1999). This calculation was performed for all samples in this study (n=29), adding a 20% uncertainty to the U_{auth} activity, and the mean $^{238}U_{auth}/^{238}U_m$ ratio was 0.77±0.18. In the $Marine_{xs}$ dose rate model, the entire detrital ^{238}U decay series is assumed to be in equilibrium, which will result in an overestimate of the true dose rate due to uranium where authigenic uranium uptake has occurred. Ages were recalculated using the revised "$Marine_{xs+auth}$" dose rate model, in which the dose rate due to $^{238}U_{auth}$ is calculated assuming equilibrium to ^{234}U, with no dose from the decay products of $^{234}U_{auth}$, and the detrital ^{238}U ($^{238}U_m$ - $^{238}U_{auth}$) decay series is assumed to be in equilibrium throughout. In the $Marine_{xs+auth}$ dose rate model, all other components are calculated as in the $Marine_{xs}$ dose rate model. Coarse silt OSL ages calculated using the $Marine_{xs+auth}$ dose rate model (Table S6) are in good agreement with the independent chronological data (Figure 3a), indicating that the age underestimation observed when using the $Marine_{xs}$ dose rate model is caused by the failure of this model to account for authigenic uranium uptake. The mean ratio of $Marine_{xs}$/ $Marine_{xs+auth}$ dose rate ages is 0.73±0.05.

Ages were calculated, using the $Marine_{xs+auth}$ dose rate model, for the 16 samples for which equivalent doses had been measured on fine quartz silt (Table S7, Figure 3b). The fine silt ages do not increase monotonically with depth, nor do they agree with the independent chronological data or the paired coarse silt ages where available (~4-6.5 m). All fine silt OSL ages are older than corresponding coarse silt OSL ages and independent age control. Together these discrepancies are taken to indicate that the dose rate model is correct, but that the fine silt equivalent doses do not represent the dose experienced since the deposition of these particles at ODP Site 658. It has been suggested that in many marine contexts coarse silt might be preferential to fine silt for dating since 90% of grains within the nepheloid (cloudy) layer in non-polar deep oceans, which results from sediment reworking, consist of grains with a diameter of 0.5-8.5 µm (Berger, 2006). Also, the $^{230}Th_{xs}$ record from ODP core 658C indicates that over the last 20 ka, this site has received 3 times more sediment laterally than vertically, though this ratio is quite variable (Adkins et al., 2006). Since 50 µm grains settle through water at ~40 times the rate of 8 µm grains, the coarse silt fraction is more likely to be incorporated via sea surface aeolian input immediately prior to deposition than is the fine silt fraction. Consequently, the discrepancy between coarse silt and fine silt OSL ages at site 658B is attributed to ocean floor reworking, without exposure to sunlight, of the latter.

4 Conclusions

Disequilibrium in the ^{238}U and ^{235}U decay series due to precipitation of insoluble isotopes from the water column is a well-known phenomenon in marine sediments (Wintle and Huntley, 1979). With appropriate $^{230}Th_{xs}$ measurements, the effect of this disequilibrium on dose rates can be accounted for (Stokes et al., 2003). At ODP Site 658, authigenic uranium uptake represents an important additional source of disequilibrium. A simple dose rate correction is proposed, which appears suitable for sediments which are much younger than the half-life of ^{234}U. However, more complex dose rate corrections will be required in older samples where significant ingrowth of $^{234}U_{auth}$ decay products has occurred. It is probable that authigenic uranium uptake in marine sediments could

disguise quite large $^{230}Th_{xs}$ activities where secular equilibrium is diagnosed on the basis of $^{226}Ra/^{238}U$ activity ratios determined using high-resolution gamma spectrometry (e.g. Jakobsson et al., 2003). Consequently, it is prudent to calculate authigenic uranium uptake for all samples when dating open ocean sediments.

Coarse silt (40-63 μm) OSL ages from site 658B are internally consistent and in good agreement with independent age control. Conversely, fine silt (4-11 μm) yielded older ages for all samples, and dates did not increase monotonically with depth. It appears likely that seafloor reworking of fine silt caused these ages to be unrepresentative of the timing of sediment formation at the sampled position. This result potentially limits the applicability of OSL dating to marine sediments, since the coarse silt component at site 658B results from its location under the North African summer dust plume. However, since the deep ocean nepheloid layer contains little material >10 μm, it is possible that accurate ages for sediment formation could be obtained from silts only slightly coarser than the 4-11 μm fraction measured here.

5 Acknowledgements

The measurements presented in the paper were funded by NERC grant NER/T/S/2001/01236 and NERC Radiocarbon Analysis Allocation No. 1036.0403, both awarded to Stephen Stokes. Calcium carbonate analysis and foraminifera picking for ^{14}C and $\delta^{18}O$ measurements was carried out by Chris Beer and Abigail Stone. Jenny Kynaston drew Figure 1. Andreas Lang is thanked for providing a constructive review. This work was initiated by Stephen Stokes, to whom the paper is dedicated.

Figures

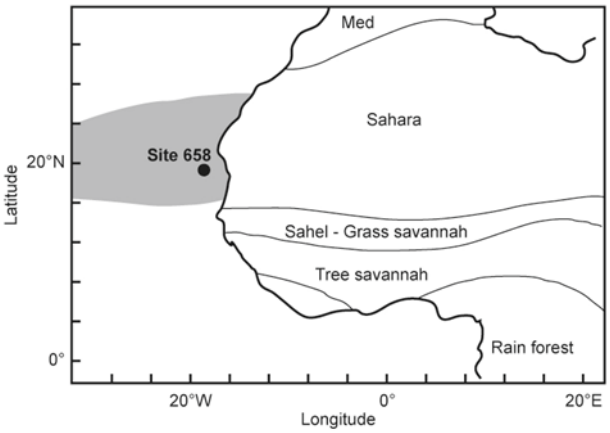


Figure 1: Location of ODP Site 658 under the summer African dust plume (grey). Redrawn from deMenocal et al. (2000).

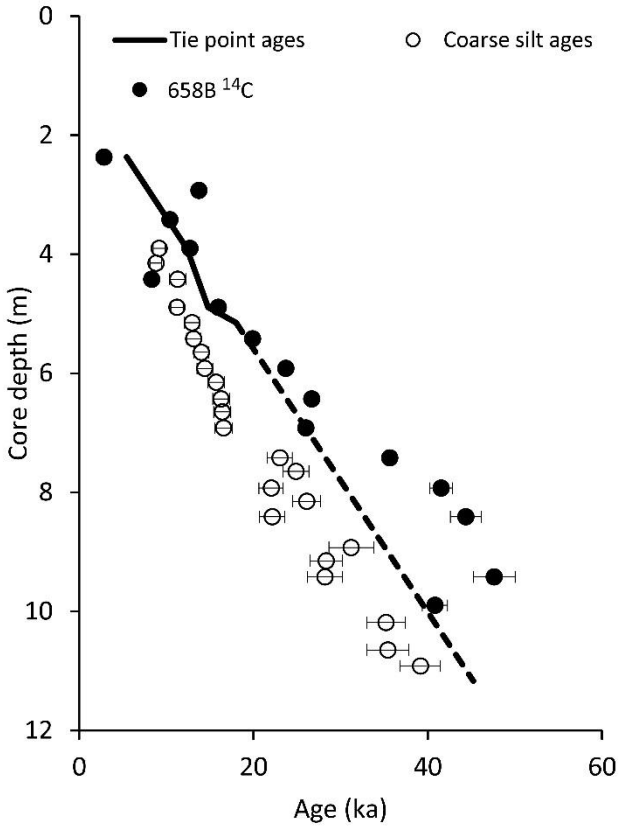
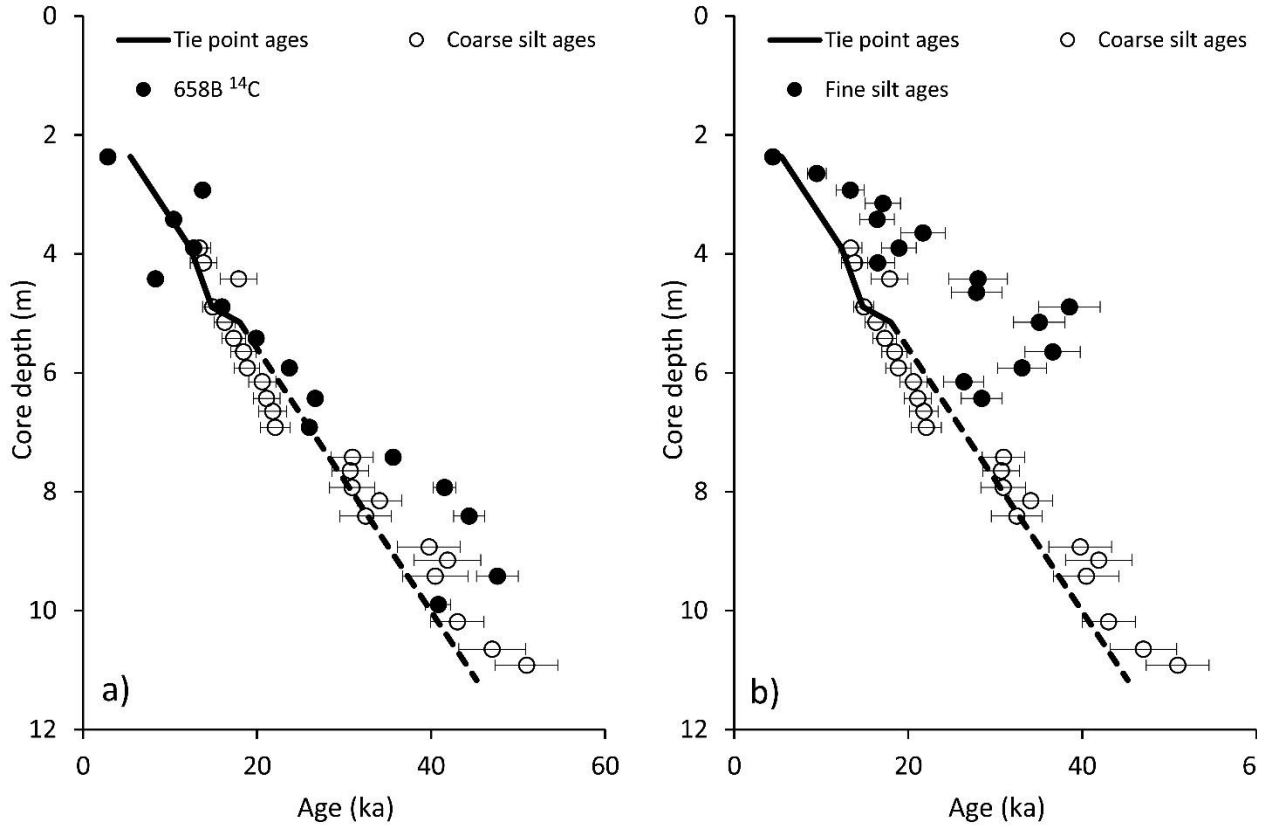


Figure 2: Chronological models for Core 658B. Filled circles represent independent age estimates determined using AMS ^{14}C on foraminifera from Core 658B and the solid line represents an age-depth relationship determined by matching stratigraphic events to external dated stratigraphies, assuming constant sedimentation between these tie points. The dashed line represents the age-depth relationship beyond 18 ka, assuming that the 22 cm/ka sedimentation rate observed between 2.37 and 5.15 m core depth continues. Open circles represent coarse silt OSL ages calculated using the $Marine_{xs}$ dose rate model.



269 Figure 3: Independent age estimates and OSL ages for Core 658B. a) Independent age estimates and
270 coarse silt OSL ages calculated using the $Marine_{xs+auth}$ dose rate model. b) Coarse silt and fine silt
271 OSL ages calculated using the $Marine_{xs+auth}$ dose rate model, plotted alongside the independent age-
272 depth model. Core 658B AMS ^{14}C ages have been removed from panel b for clarity.
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