Quartz Age Extension Applied to SE Asian Cover Sands

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Abstract

A significant feature of the surface sediments of southeast Asia is a regionally extensive layer of distinctive red, quartz-rich, cover sand observed throughout Vietnam, Cambodia, Laos and Thailand, and further afield. In many locations, these cover sands immediately overlay a laterite layer containing tektites, known as the Muong Nong type, associated with a large meteorite impact between 750 to 800ka in Indochina. Sections of these cover sands at sites in Thailand, Laos and Vietnam have been investigated using field and laboratory profiling, and quartz SAR procedures. In some locations the sections consist of a layer of low-sensitivity quartz with saturated signals overlain by a visibly indistinguishable layer of high-sensitivity quartz with ages less than c. 35ka. Further work has been undertaken to attempt to extend quartz luminescence dating for the older materials, including samples associated with the tektites, using thermally stimulated or transferred luminescence to access traps that are expected to saturate at higher doses. Luminescence was recorded during sample heating and hold, giving thermoluminescence (TL-ramp) and isothermal decay (ID) data, in addition to optically stimulated luminescence after the transfer (Thermally Transferred Optically Stimulated Luminescence, TT-OSL) measurements. These measurements have produced equivalent dose values of up to 250Gy, and ages of 70-125ka, for these older materials, which is significantly younger than would be expected from the association with the tektites. Investigation of the traps associated with these signals has produced properties consistent with prior investigations, suggesting that these are not sufficiently stable at environmental temperatures above 25°C to permit age extension using these methods.

Keywords Thermally Transferred Optically Stimulated Luminescence; Thermoluminescence; Isothermal Decay

Highlights

- TT-OSL applied to coarse quartz grains from SE Asia
- Ages obtained significantly less than assumed age of associated tektites
- Thermal stability of TT-OSL traps assessed
- Thermal stability limits age range to less than 800ka at temperatures above 25°C

1. Introduction

A feature present across large parts of south east Asia (including southern China, Laos, Vietnam, northern Cambodia and north east Thailand) is a distinct layer of sand. These cover sands are generally red-coloured, consisting of virtually mono-mineralic quartz sand and in undisturbed locations can be between 3 and 15m thick. These deposits often lie directly upon the weathered, brecciated or lithified basement rocks or above a distinct gravel layer. The origin of these sands is unclear, having been variously attributed to lacustrine, marine, colluvial, fluvial, aeolian or biomantle processes. The literature supporting these different models is summarised in Sanderson et.al. (2001), where data obtained from Khon Kaen in NE Thailand with broadly concordant TL and OSL ages in the last 50 ka was interpreted as supporting aeolian origins for the cover sands. Recent investigation near Huai Om in Thailand identifies air-fall deposits lying above base-surge deposits in the cover sands as the result of a significant regional Quaternary meteorite impact (Tada et al., 2020).

The cover sands are associated with another regional feature; an extensive area of tektite deposition associated with the impact event centred over SE Asia 750-800ka BP (see, for example, Schwarz et al., 2016; Jourdan et al., 2019; Michel et al. 2021). The tektite field covers an area of southern Asia and Australasia of approximately 8000 x 13000 km, from Madagascar into the Pacific Ocean, southern China to all of Australia and potentially into Antarctica (see, for example, Whymark, 2021). In SE Asia, lateritic granule layers within the lower section of the cover sands contain tektites from this impact, with many of them appearing to be undisturbed since deposition (Tada et al, 2020). The details of the stratigraphic relationships of the cover sands with the underlying gravels and tektites will be presented in a future paper, but are summarized within Figure 1. In the work presented here, the optically stimulated luminescence properties of the cover sands are reviewed and a potential method, using thermal transfer techniques, to extend the age range of quartz OSL to cover the expected age of the tektites is evaluated.

1.1 Sample locations

Data from investigations conducted for the tektite studies, and earlier studies where OSL dates and profile measurements have been collected, have been combined to characterise the general optically stimulated luminescence properties of the cover sands. The locations of these samples are shown in Figure 2.

The samples from Khon Kaen (six samples between 20-200cm depths, reported by Sanderson et.al., 2001), Krahad (three samples from Unit 3 between 1.1-1.8m, reported by Porat, 2017), Kok Yai (three samples from Unit 3 between 0.8-1.7m reported by Porat, 2017, and two further samples from the Unit 2, the meteorite impact blast granule layer, and 1m above this (Unit 3) report by Cresswell et.al., 2019a) and Sa Kaeo (one sample from Unit 2 reported by Cresswell et.al., 2019a) all show similar properties, with sensitive quartz (>10^5 cGy^{-1}) and SAR-OSL ages in the 10-35ka range. Sediment profiles from these sites generally show the same simple stratigraphy with depth. In contrast, samples from Huai Om (two OSL samples from the base of the cover sand at a depth of 210cm and the top of the ejecta layer at a depth of 295cm, plus 25 profile samples from 160-390cm depth reported in Cresswell et.al., 2019a,b) and Pakse (one sample from between two possible ejecta layers reported in Cresswell et.al., 2019b) contain quartz with much lower sensitivity (<5000 cGy^{-1}) and the natural signals exceed the SAR-OSL saturation limit (>50ka for these samples), the Huai Om profile shows greater quartz sensitivity (comparable with the other sites) for the top sample at

160cm depth, with a reduction in sensitivity of more than an order of magnitude for samples at 170cm depth and deeper. The basal sample from Hue (collected at 210cm depth from a profile of 21 samples between 10-210cm depth reported in Cresswell et al., 2018a,b) shows a mixture of both younger, sensitive quartz and older, insensitive quartz characteristic of both these groups of sites. The Tad Huakhon sample (Cresswell et.al. 2019b) was collected from below a currently undated lava flow and contains quartz that has high sensitivity with natural OSL signals in excess of the saturation limit.

Assuming that these results from eight locations reflect regional patterns, they strongly suggest that these apparently similar cover sands represent at least two different origins or depositional processes. The upper layers are characterised by higher sensitivity quartz, with profiles showing increasing OSL age with depth, with the samples reported here giving ages in the 10-35ka range, consistent with aeolian deposition processes. The lower layers are characterised by quartz with sensitivities at least two orders of magnitude lower than the upper layers, and OSL dates that exceed the age range of the SAR-OSL method. The working model that will be explored further in a future publication, summarised in Figure 1, is that these materials are consistent with deposition by base surge following the meteorite impact and subsequent air-fall processes, with later aeolian deposition of sediments from a distant source.

1.2 Dating challenge

The tektites are associated with the lower layers of the cover sands which are older than the SAR-OSL limit, which are virtually mono-mineralic quartz and hence luminescence dating using feldspars is not an option. Thermally Transferred Optically Stimulated Luminescence

(TT-OSL) has been proposed as a method to access deeper traps in quartz, which are expected to saturate at higher doses than the traps accessed by OSL. This approach has been explored for these samples, and in this paper the approaches to use thermal transfer measurements are described and the question of thermal stability at the ambient temperatures of these locations addressed.

Several different protocols for TT-OSL have been used in previous studies (e.g.: Wang et.al. 2006a, b; Adamiec et.al. 2010; Shen et.al. 2011; Thiel et.al. 2012; Brown and Forman 2012; Chapot et.al. 2016), but these all have common elements. The OSL from the sample is measured as normal, then the sample is heated to an elevated temperature and kept there to allow transfer of charge from deeper traps to the OSL accessible traps, and then the amount of charge transferred is measured by a repeat OSL measurement (this is the TT-OSL measurement). TT-OSL signals are typically 1-2% of the initial OSL. TL signals can also be recorded during the ramp to the elevated temperature (TL-ramp) and during the hold phase (isothermal decay – ID). Costello (2009) noted significant differences between quartz from different locations through LM-OSL measurements, and noted that ID (which was described as phosphorescence during the thermal transfer process) is a useful analytical approach to understanding the processes. Post-OSL TL approaches, where a TL readout follows the OSL measurement, also has been used previously to extend the dose response, with TL signals in the 300-350°C region shown to correlate with dose (Kinnaird et.al. 2010).

Aitken (1998) proposed a model of two forms of transfer based on measurements of optical bleaching at room temperature, termed ‘recuperation’ where electrons present in the OSL traps are transferred to ‘refuge traps’ from where they can be transferred back to the OSL traps, and ‘basic transfer’ where electrons in hard to bleach traps are transferred to the OSL traps during preheating. Wang et.al. (2006a,b) demonstrated TT-OSL from fine grain (4-11µm) quartz from Chinese loess, using 270s optical stimulation to completely remove fast and medium OSL components, with heating at 260°C for 10s followed by repeat OSL measurement. The resulting TT-OSL was observed to have two components attributed to recuperation (ReOSL) and basic transfer (BT-OSL), with a measurement sequence proposed to separate the BT-OSL and ReOSL from the total TT-OSL. Normalised ReOSL dose response curves continued to grow beyond 4kGy doses with D_e values with ~20% uncertainties, allowing the calculation of dates beyond 1Ma. Adamiec et.al. (2008) reported experimental data supporting an alternative model for the ReOSL signals as being a single transfer from traps with similar thermal stability as the fast OSL traps but only emptied optically by long exposure, compared with the double transfer mechanism with refuge traps. This single transfer model is then more similar to the BT-OSL process. Adamiec et.al. (2010) explored the properties of these traps, again using fine (4-11µm) grains, proposing a protocol with a 260°C 10s preheat, 200s LM-OSL at 125°C, a thermal transfer at 260°C for 10s and 100s CW-OSL measurement of the TT-OSL. Shen et.al. (2011) applied a modification of the procedure of Wang et.al. (2006a) with transfers 300°C to different size fractions, showing no significant difference in TT-OSL response as a function of grain size. Thiel et.al. (2012) used a protocol proposed by Stevens et.al. (2009) (60s OSL measurements, a 260°C 10s transfer and a thermally assisted optical bleaching to remove residual trapped charge (400s OSL at 280°C) prior to adding regenerative doses) on sand-sized (180-250µm) quartz grains, showing that deposits older than 250ka showed an age underestimation by TT-OSL compared to pIRIR measurements of K-feldspar from the same sample, attributed to thermal instability in the hard to bleach traps accessed by TT-OSL. Brown and Forman (2012) adapted the Stevens et.al. (2009) protocol by reducing cutheat and increasing the bleaching temperature to 350°C, applied to fine grain loess. Chapot et.al. (2016) modify the procedure of Adamiec et.al. (2010) using a 300s OSL rather than 80s LM-OSL, on fine grain loess.

In this paper, appropriate protocols are explored for use with the coarse grain quartz of the lower sedimentary layers of the cover sands of south-east Asia, expanding on the prior work summarised above. And, the thermal stability of the harder to bleach traps associated with these thermally transferred signals is examined at temperatures in excess of the environments of these previous studies.

2. Methods

2.1 Exploratory Analyses

As noted above, prior studies using TT-OSL have used different protocols for measurements, in some instances adjusting the protocol to the samples being analysed (for example Brown and Forman 2012). Some studies have also indicated variations in the parameters for the TT-OSL source trap. Therefore, an exploratory analysis using two aliquots of quartz from Hue, with natural signals exceeding the OSL saturation limit, was conducted with different hold times (between 10 and 60s) and temperatures (between 240°C and 360°C) to determine if any adjustments are needed to the protocol, and identify any significant differences in trap parameters in these samples, compared to prior literature. It has already been shown that post-OSL TL extends the dose range (Kinnaird et.al. 2010), in this work the use of lower temperature TL is also examined. So, the luminescence signals generated during the heating to transfer temperature, and hold at that temperature, are also explored as an option for recovery of equivalent doses from the TT-OSL source trap.

The counts recorded during the TL-ramp, the isothermal decay (ID) during the hold and the TT-OSL were used to identify the optimal protocol that maximises these signals for the sample investigated. All measurements were conducted using a Risø DA-15 system, with the use of low-level commands to enable measurements of photon emission during the ramp and hold (TL-ramp and ID) without lowering the lift between these. The measurement steps are outlined in Table 1; note that set A corresponds to the procedure of Adamiec et.al. (2010) except for the use of CW OSL prior to the thermal transfer, with the "natural" dose for sets A-D being any residual signal left after prior measurement cycles with these sets not including the low-level commands (i.e.: the lift was lowered after the TL-ramp and raised again for the hold). Figure 3 shows the TL-ramp and hold (ID) for sets E-H, where the ID measurement follows immediately after the TL ramp, where it can be seen that the TL peak for these samples is at about 300°C. The integrated counts for the TL-ramp and OSL measurements, and the count rate for the ID, are given in Table 2. For a transfer temperature of 260°C, the TT-OSL to OSL ratio is similar to that reported previously, but for higher transfer temperatures the TT-OSL is very small with only the 30s hold at 280°C giving a TT-OSL response. Thus, for temperatures above 280°C the OSL traps do not retain any transferred charge and no TT-OSL is produced, though at these temperatures data from the transfer is recorded in the TL-ramp and ID signals. A protocol with a TL-ramp to 260°C held for 30s maximises the TL-ramp, ID and TT-OSL signals in the quartz from Hue used for these exploratory analyses, and so will be used in subsequent analyses of the other quartz samples in this study.

2.2 Application to SE Asian Cover Sand Samples

To allow direct comparison between OSL and the thermal transfer (TL-ramp, ID and TT-

OSL) signals, a hybrid sequence incorporating both measurements on each aliquot was used, as shown in Table 3. This procedure initially records the natural OSL, TL-ramp, ID and TT-OSL from the aliquots. In expectation that the thermal treatments and large doses involved in the thermal transfer procedure could induce significant sensitivity changes, an OSL-SAR sequence of small (1Gy) test doses and regenerative doses to 50Gy was then applied before thermal transfer. After completion of the OSL-SAR sequence, a sequence of measurements of the thermal transfer signals following regenerative doses from 50Gy to 1000Gy was conducted. Thus, for aliquots which produce a quantified SAR-OSL equivalent dose this can be directly compared with the equivalent dose produced by the three thermal transfer measurements. Samples were measured in sets of 16 aliquots, divided into four sets with different pre-heat temperatures used for the OSL measurements. In practice, the thermal transfer signals from the 50Gy test dose were too small to allow normalisation of these signals and so un-normalised TT-OSL and TL-ramp signals were used. This sequence was applied to samples collected from the basal layers of the cover sands at Hue, Huai Om, Pakse, Sa Kaeo and Tad Huakhon.

3. Results

A typical set of dose response curves for the three thermal transfer measurements is shown in Figure 4, for a sample from Huai Om. The ID and TL-ramp signals produced during the thermal transfer process are the largest signals, however they both show response curves that are saturating at 600-1000Gy, and the ID natural signals are highly dispersed with many exceeding the saturation value for the response curve. The TT-OSL signals are smaller, but with a response curve that is only beginning to fall below linear to 1000Gy, with natural signals that are tightly grouped.

The measurement sequence was applied to samples from the upper layers of cover sand, which had produced well constrained OSL dates, to allow comparison between equivalent dose determinations using the thermal transfer approach to the SAR-OSL equivalent doses for the same aliquots. Three samples were used, two from Kok Yai and one from Sa Kaeo (Cresswell et.al. 2019a), with equivalent dose values from the SAR-OSL of 4.3 ± 0.1Gy (SUTL2990, Sa Kaeo), 10.7 ± 0.5Gy and 8.6 ± 0.1Gy (SUTL2987 and 2988, respectively, Kok Yai). The data for each aliquot are shown in Figure 5, with linear regressions shown as a guide. The precision of the data, in particular for the thermal transfer values, are low with significant scatter. It is, however, clear that the slopes of regressions are significantly less than one and that the thermal transfer methods significantly underestimate the E_D. It is also evident that the ID data are more scattered with a large intercept and large uncertainties on the regression fit. Given the poorer comparison to known age samples and greater scatter in natural signals from the ID measurements, the TL-ramp and TT-OSL measurements are preferred for these samples.

When applied to the samples from the basal layers of the cover sands, which are associated with the tektites, the E_D values determined range from 100Gy to 250Gy (with uncertainties of approximately 20% on the E_D for each sample), corresponding to ages of 70-125ka (for measured dose rates mostly in the range of 1-2mGy a^{-1}). These are significantly younger than the expected ages due to the association with the tektites. It is noted that the ambient temperature below 2m at the sampling locations is 26-28°C, which raises the possibility that the trap accessed by the thermal transfer processes is unstable at these temperatures, hence resulting in an underestimation of the age based on these measurements.

3.1 Trap Stability

The properties of the traps associated with TT-OSL have been investigated by several authors. Faershtein et.al (2018) reviewed values reported by Li and Li (2006), Adamiec et.al. (2010), Shen et.al. (2011), Brown and Forman (2012) and Chapot et.al. (2016) giving an average E of 1.36 ± 0.45 eV and log_{10}s of 10.4 ± 3.6 log_{10} s^{-1}, with calculated trap lifetimes at 10°C assuming first order reaction kinetics of between 10^5 and 10^9 years (average $10^{6.6 \pm 1.5}$), and for Mediterranean sands they analysed E values of 1.4-1.6eV (average 1.50 ± 0.06) and log_{10}s of 11.9-13.7 log_{10} s^{-1} (average 12.8 ± 0.6). Thiel et.al. (2012) measured sands from Tunisia with an environmental temperature of 19°C, and observed a TT-OSL age underestimation compared to pIR-IR ages from which a mean lifetime of 0.69Ma was determined for the traps associated with TT-OSL, which was compared with lifetimes calculated from the parameters of Adamiec et.al. 2010 which predicts a lifetime of 0.71Ma at 19°C. Prior literature suggests that for environmental temperatures up to 20°C the traps associated with TT-OSL should have lifetimes of in the 10^5-10^6a range.

The TL-ramp and ID measurements can be used to determine the properties of the TL-trap for the samples analysed in this work, which follows the approach of Costello (2009). It is noted that these are not high quality precision measurements of these parameters, but sufficient to determine whether the properties are consistent with previously reported values. The trap depth can be determined from the TL-ramp measurements, from the slope of an Arrhenius plot at the rise of the TL signal. Figure 6 shows such a plot for a single aliquot, with data plotted assuming the nominal temperature reported by the Risø reader is accurate (black line) and for a ±10K deviation from this (red and blue lines). Across eight aliquots used in the exploratory measurements (two at each of four pre-heats) the mean value determined is 0.96

± 0.08eV, which after correction for thermal quenching (0.64eV) (from Wintle, 1975) gives a trap depth of 1.60 ± 0.10eV. The fast component of the ID is then used along with this trap depth to estimate the associated frequency factor for the trap, giving a result of 1.2×10^{13} to 9.3×10^{14} s^{-1}. The trap depth and frequency factors are consistent with the values previously reported, as summarised above, with the frequency factor at the top end of the range of previously reported values.

The lifetime for a trap can be calculated from the trap depth (E), frequency factor (s) and temperature (T, in K) as $t = e^{E/kT}/s$. These values were calculated for three trap depths corresponding to the range of values calculated above (1.5, 1.6 and 1.7eV) and for the corresponding frequency factors rounded to one significant figure (0.1, 1.0 and 10 $\times 10^{14}$ s^{-1}) for temperatures ranging from 0 to 40°C, as shown in Table 4. It can be seen that in the 25-30°C range, typical of the ambient temperatures for deep soils in the region, the lifetimes calculated for these three trap parameters are in the range of 20ka to 2Ma. For most of these trap parameters at these temperatures, the calculated lifetime is short compared with the 750-800ka age of the tektites. Thus, these traps may experience thermal fading at the ambient temperatures of the region, and the calculated E_D and age values for these samples may be underestimated.

4. Discussion and Conclusions

Portable and laboratory luminescence profile measurements have shown a cryptostratigraphy within SE Asian cover sands, with younger strata (10-35ka) characterised by sensitive quartz and older strata characterised by low sensitivity quartz with natural signals in excess of SAR saturation (>50ka) (Cresswell et al., 2018a, b; Cresswell et al., 2019a, b). This stratigraphy suggests that there are multiple sources and processes for the development of these features, with a change in sediment source and/or process no earlier than 35ka. The origins and formation processes of these cover sands pose interesting questions, which would require the collection of samples targeting this boundary. However, the focus of the work reported here is to explore options for dating the older strata that exceed the limits of SAR-OSL.

Exploiting charge in deeper quartz traps through thermal stimulation or transfer processes should allow an extension of E_D determination beyond the saturation limits of OSL measurements. Previous studies have shown the potential of TT-OSL to extend ages beyond 1Ma. Here an exploratory analysis using a small number of aliquots has determined that a ramp to 260°C and hold for 30s results in the largest TL-ramp, ID and TT-OSL signals. Comparison between E_D values determined by these methods and by SAR-OSL on younger samples, where there were aliquots that did not saturate, show that both TL-ramp and TT-OSL methods produce an approximate linear relationship with the SAR-OSL, through data with significant scatter and low precision, whereas the ID measurements on these younger samples produce a highly scattered relationship with inconsistent results. Therefore, the TL-ramp and TT-OSL measurements were used for measurement of the older materials.

For the samples analysed in this study, dose extension gives E_D values of 100-250Gy (corresponding to ages of 70-125ka), which are significantly younger than assumed age of the associated tektites within these lower layers of the cover sands. Estimates of the energy level and frequency factors of these traps suggest that they are not stable over the required 0.5-1.0Ma age range for dating sediments associated with tektites from an impact at 750-800ka BP, at temperatures typical in SE Asia. For dating sediments of this age, which is also relevant to the Brunhes-Matuyama geomagnetic reversal at a similar age, in these environmental conditions a trap life time of approximately 20Ma would be needed to minimize the requirement to use a significant correction for trap stability. For frequency factors in the range of 10^{13} to 10^{15} s^{-1}, this would require a trap depth of at least 1.67-1.79eV. Alternative methods of extending the dose range for quartz luminescence, for example photo-transferred TL (PTTL), thermally assisted OSL (TA-OSL) or violet stimulated luminescence (VSL) may access traps that are more thermally stable, and thus be applicable to materials from locations where sediments are exposed to temperatures in excess of 20°C. These alternative approaches could be considered for the materials analysed here. The dose extension methods described here should be applicable to at least 500Gy (TL-ramp or ID) or 1kGy (TT-OSL) in more temperate locations, where temperatures rarely exceed 20°C.

Acknowledgements

Carling benefited from a Leverhulme Emeritus Fellowship (2016–2018) and from support in the field provided by Prof. R. Tada (Japan Society for the Promotion of Science (JSPS) KAKENHI Fostering Joint International Research (B) 18KK0092 (2018-2021)). The luminescence analyses were part funded by the Biotechnology and Biological Sciences Research Council under a Global Challenges Research Foundation Award for Global Agriculture and Food Systems Research (BB/P022693/1) awarded to Darby.

This paper is dedicated to the memory of Paul Bishop, whose long standing interest in SE Asia initiated this work.

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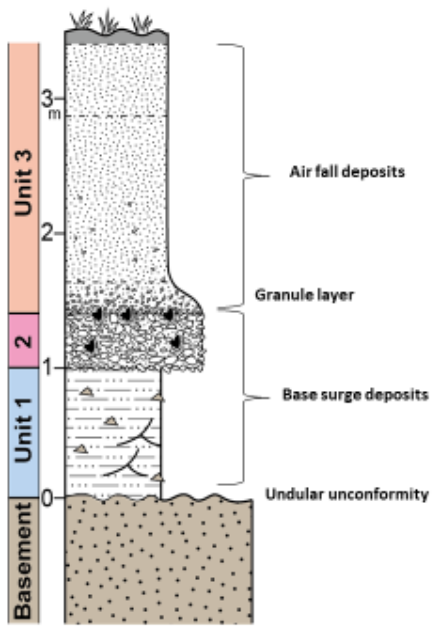


Figure 1: Summary depositional sequence of meteorite impact deposits in south-east Asia. The basement is undefined as it can vary across the region. The thicknesses of Units 1 and 2 as shown are typical, although Unit 3 can be several metres thick. Unit 1 is commonly laminated fine to coarse angular sand beds containing bedrock chips, local cross-bedding, and the bedding grain-size varies non-systematically upwards. Unit 2 consists of unstratified to weakly stratified, well-rounded white quartzite pebbles with frequent (black) tektites. Unit 3 is unstratified (i.e. massive) fine to coarse sands.

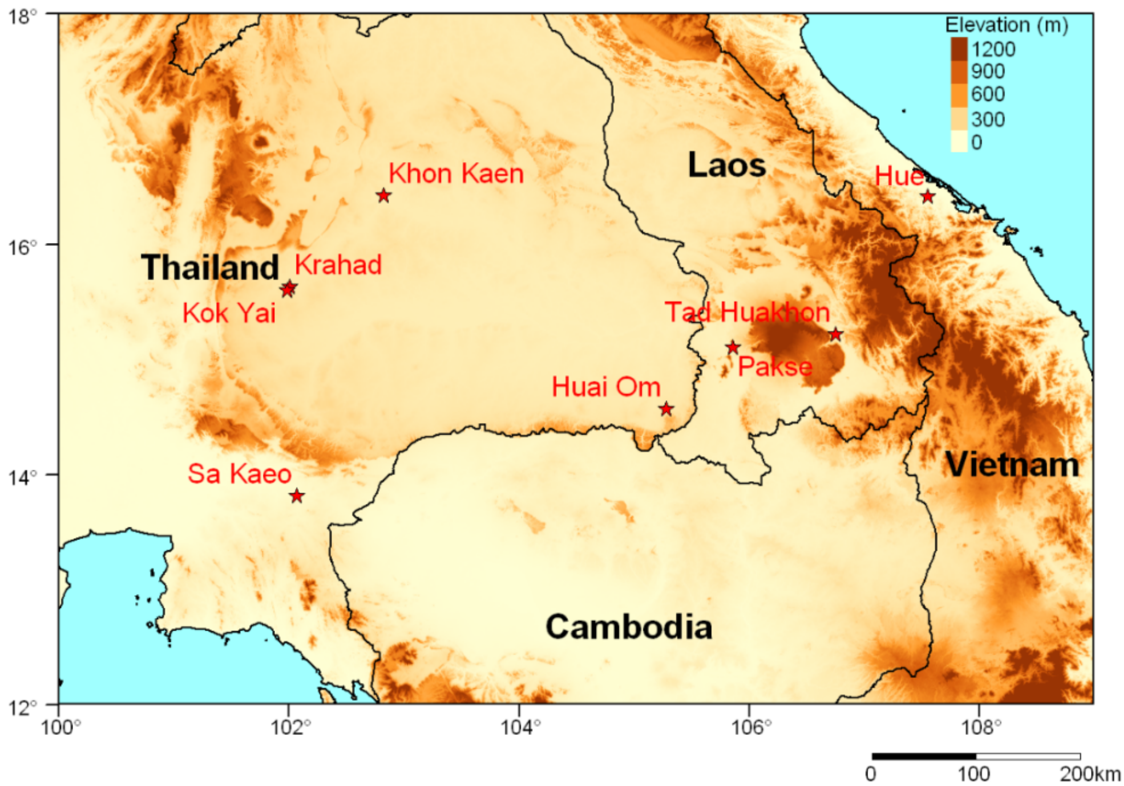
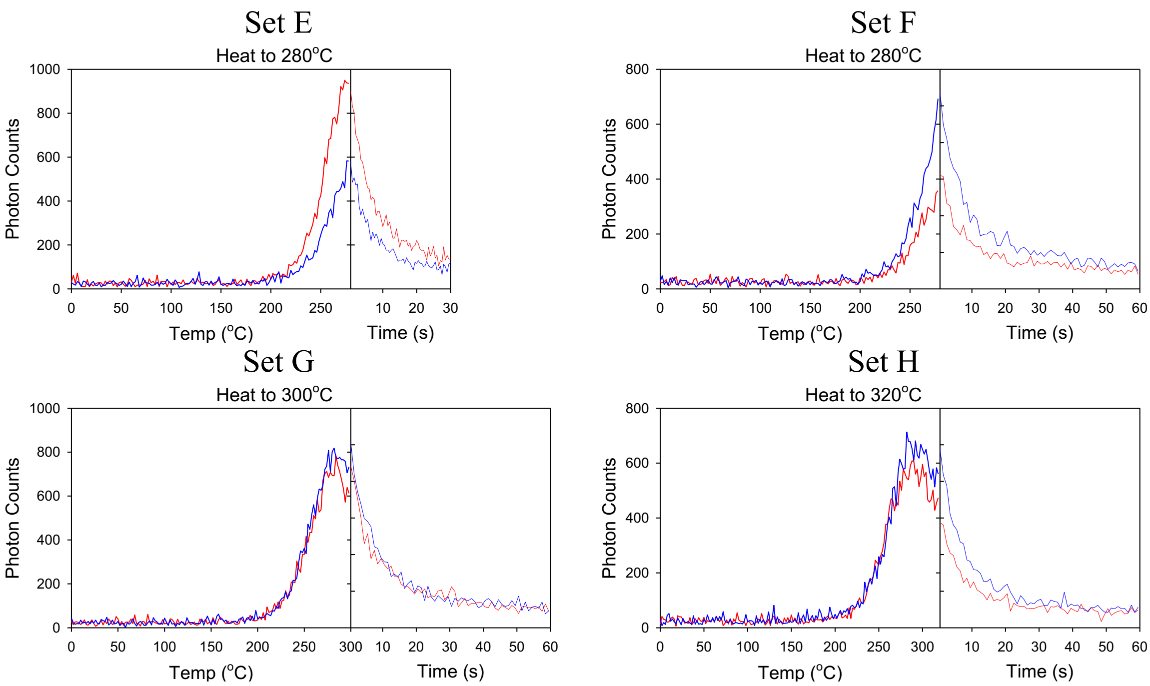


Figure 2: Locations of luminescence measurements of cover sands in SE Asia: Khon Kaen (Sanderson et.al. 2001); Kok Yai (Porat 2017, Cresswell et.al. 2019a); Krahad (Porat 2017); Hue (Cresswell et.al. 2018a,b); Sa Kaeo (Cresswell et.al. 2019a); Huai Om (Cresswell et.al. 2019a,b); Pakse & Tad Huakhon (Cresswell et.al. 2019b).



459 Figure 3: TL-ramp and ID (hold) for exploratory analysis of two aliqouts, sets E-H of Table 2.

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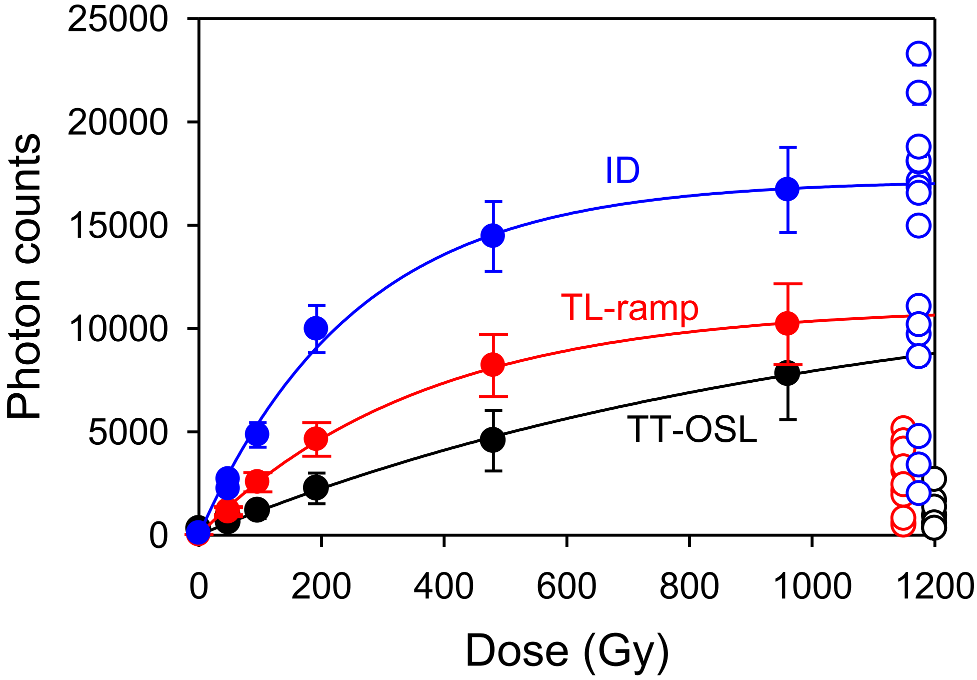

Figure 4: Dose response curves for the three thermal transfer methods, from a sample from Huai Om. The open circles on the right hand side indicate the photon counts from the natural signals for each measurement. ID measurements in blue, TL-ramp in red and TT-OSL in black.

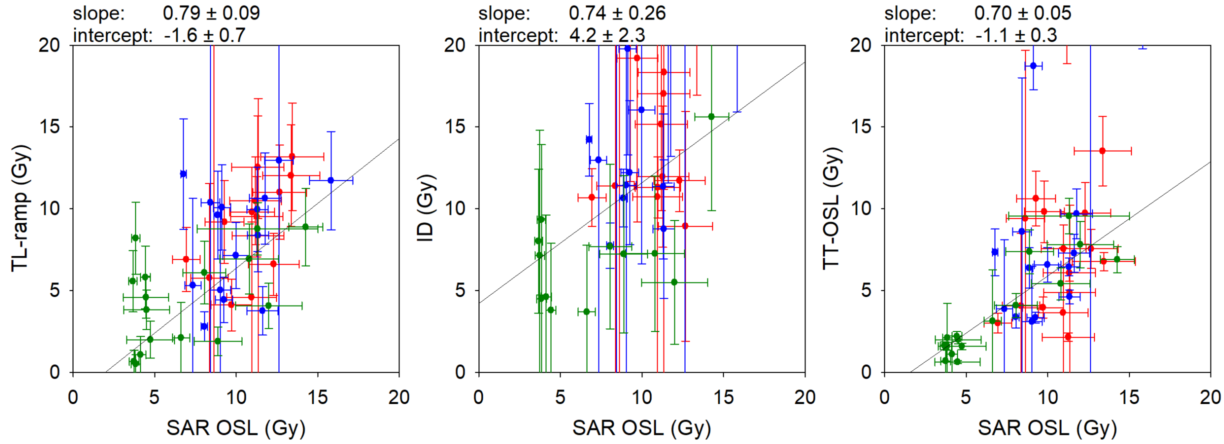

Figure 5: Comparison between TL-ramp, ID and TT-OSL equivalent doses with the SAR-OSL equivalent doses from the same aliquot, for younger samples from the upper layer of the cover sands. Data from 16 aliquots of each of two samples from Kok Yai (blue, E_D=8.6 ± 0.1Gy, and red, E_D=10.7 ± 0.5Gy) and a sample from Sa Kaeo (green, E_D=4.3 ± 0.1Gy).

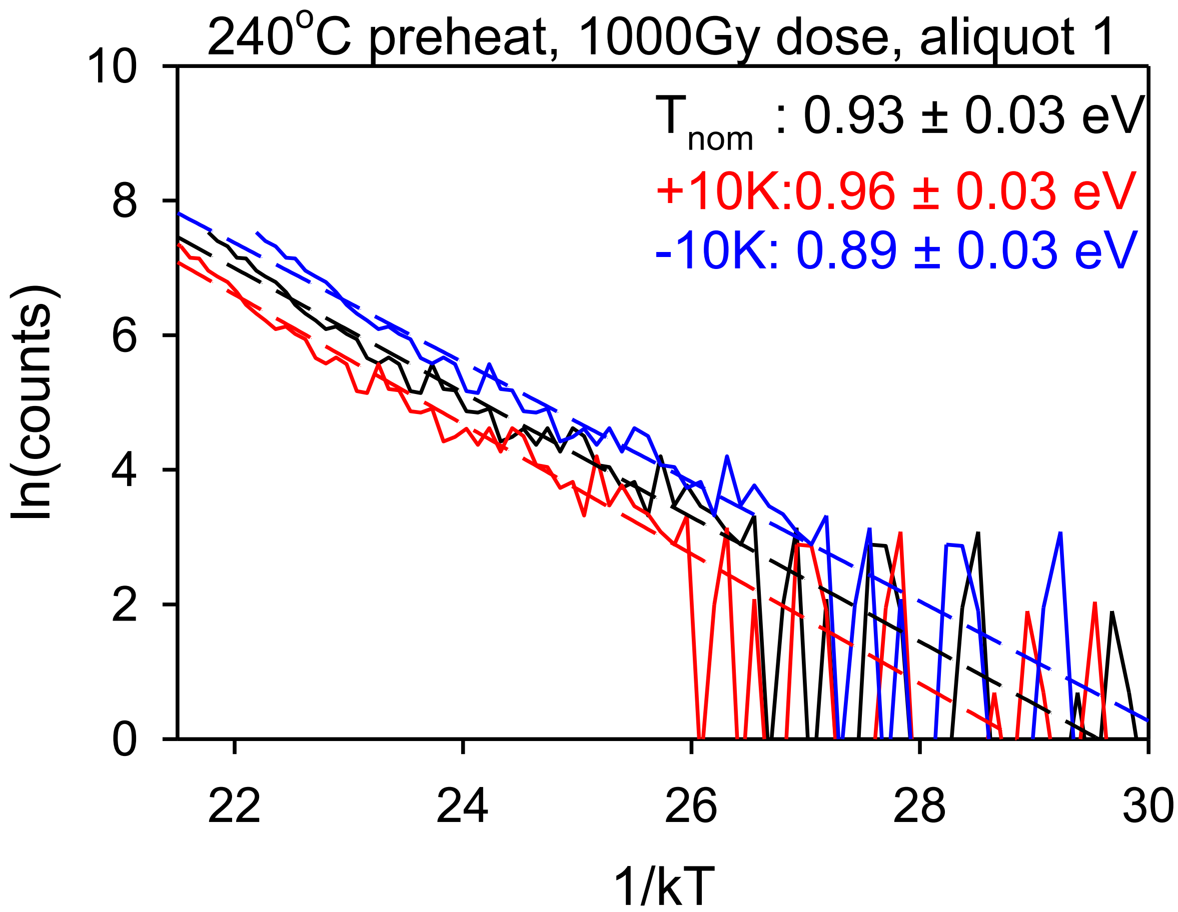

Figure 6: Example Arrhenius plot for one of 8 aliquots, showing the rising slope of the TL-ramp signal and associated trap depth value, assuming the nominal temperature from the Risø reader is accurate (black) and a ±10K deviation (red and blue).

Table 1: Exploratory TT-OSL procedure. Set A corresponds to the procedure of Adamiec et.al. (2010).

Step	Set A	Set B	Set C	Set D	Set E	Set F	Set G	Set H
1	Dose ("natural", 100Gy, 200Gy, 500Gy, 0Gy)				Dose (200Gy)			
2	PH 260°C 10s				PH 220°C 10s			
3	OSL 100s at 125°C				OSL 100s at 125°C			
4 - transfer & ID	PH 260°C 10s	PH 260°C 30s	PH 280°C 10s	PH 280°C 30s	PH 280°C 30s	PH 280°C 60s	PH 300°C 60s	PH 320°C 60s
5 – TTOSL	OSL 100s at 125°C							
6	TD (5Gy)							
7	PH 220°C 10s							
8	OSL 100s at 125°C							
9	Thermal treatment 350°C 200s							
10	Go to step 1				-			

Table 2: Counts for the initial OSL, TL-ramp and TT-OSL measurements for the exploratory analyses, with count rates for the ID and ratio of TT-OSL to OSL percentage.

Set	Aliquot	OSL	TL-ramp	ID cps	TT-OSL	TT-OSL : OSL %
A	1	17373 ± 146	15171	365 ± 6	354 ± 61	2.0 ± 0.4
	2	11355 ± 121	8370	228 ± 5	101 ± 58	0.9 ± 0.5
B	1	5521 ± 92	5343	139 ± 2	121 ± 52	2.2 ± 0.9
	2	35451 ± 198	8509	203 ± 3	-40 ± 60	-0.1 ± 0.2
C	1	6634 ± 102	11582	388 ± 6	250 ± 57	3.8 ± 0.9
	2	42317 ± 216	12096	464 ± 7	286 ± 60	0.7 ± 0.1
D	1	8755 ± 112	8592	275 ± 3	121 ± 57	1.4 ± 0.7
	2	23100 ± 164	8844	313 ± 3	55 ± 56	0.2 ± 0.2
E	1	22280 ± 170	18000	334 ± 9	287 ± 63	1.3 ± 0.3
	2	10760 ± 120	11007	189 ± 7	129 ± 57	1.2 ± 0.5
F	1	6120 ± 100	7888	67 ± 2	0 ± 56	0 ± 0.4
	2	30310 ± 190	10976	122 ± 2	190 ± 60	0.6 ± 0.2
G	1	20230 ± 160	20964	105 ± 2	-78 ± 54	-0.4 ± 0.3
	2	38650 ± 210	22463	115 ± 2	76 ± 60	0.2 ± 0.2
H	1	11940 ± 130	21835	65 ± 2	-10 ± 57	-0.1 ± 0.5
	2	26030 ± 180	24111	110 ± 2	141 ± 55	0.5 ± 0.2

Table 3: Outline of sequence for OSL and thermal transfer measurements on the same aliquots. Measurements were conducted in four sets (A-D) of four aliquots with different pre-heat temperatures (220°C to 280°C).

Step	**Set A**	**Set B**	**Set C**	**Set D**
1	Dose – 0Gy for natural; regen doses of 10, 20, 30, 40, 50, 0 and 10Gy for OSL SAR; regen doses of 50, 100, 200, 500, 1000, 0, & 50Gy for dose extension			
2 – PH	PH 220°C 10s	PH 240°C 10s	PH 260°C 10s	PH 280°C 10s
3 - OSL	OSL 60s at 125°C (all measurement)			
4 - TL	TL ramp to 260°C (for natural and dose extension)			
5 - ID	Isothermal decay for 30s (for natural and dose extension)			
6 - TTOSL	TT-OSL 60s at 125°C (for natural and dose extension)			
7 - TD	1 Gy Test Dose (for natural and OSL SAR)			
8 - PH	PH 220°C 10s	PH 240°C 10s	PH 260°C 10s	PH 280°C 10s
9 - OSL	OSL 60s at 125°C (for natural and OSL SAR)			
10 - TD	50 Gy Test Dose (for natural and dose extension)			
11 - PH	PH 220°C 10s	PH 240°C 10s	PH 260°C 10s	PH 280°C 10s
12 - OSL	OSL 60s at 125°C (for natural and dose extension)			
13 - TL	TL ramp to 260°C (for natural and dose extension)			
14 - ID	Isothermal decay for 30s (for natural and dose extension)			
15 - TTOSL	TT-OSL 60s at 125°C (for natural and dose extension)			
16	Thermal treatment 350°C 200s (for dose extension)			

Table 4: Lifetimes for the trap accessed by the thermal transfer processes as a function of trap depth (E), frequency factor (s) and temperature.

E (eV)	**s (x10^{14} s^{-1})**	**Temperature (°C)**							
		0	10	15	20	25	30	35	40
1.5	0.1	13Ma	1.4Ma	470ka	170ka	62ka	24ka	9.3ka	3.8ka
1.6	1.0	100Ma	9Ma	2.9Ma	960ka	330ka	120ka	44ka	17ka
1.7	10	825Ma	64Ma	19Ma	6Ma	1.9Ma	645ka	225ka	80ka