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Challenges involved in obtaining luminescence ages for long records of aridity: examples from the Arabian Peninsula

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Abstract

In the last 35 years luminescence dating has provided a large database of ages for the deposition of dunes in desert environments. Over 380 ages have been generated for dunes from the Arabian Peninsula and demonstrate episodic dune deposition in the late Quaternary, but give a less clear pattern prior to ~50 ka. Interpreting databases of luminescence ages faces two issues. First, the precision of luminescence ages in the last 50 ka is between 5 and 10%, but for older ages the uncertainties may be much larger as luminescence signals reach saturation. Second, different luminescence signals from quartz and from feldspar have been used over the last 35 years as the method has developed and expanded. These different signals have different saturation limits and different rates at which they are reset by exposure to daylight at deposition. Approaches which focus on the most light sensitive signals (e.g. quartz OSL) are better suited to dating recent events, while those which show growth of the luminescence signal over the largest dose range (e.g. the thermally transferred OSL (TT-OSL) signal from quartz and the post-infrared infrared stimulated luminescence (pIR-IRSL) from feldspars) have the potential to date much older events.

Keywords: Luminescence; OSL; IRSL; TT-OSL; desert dunes; limits

1. Introduction

Luminescence dating methods have revolutionised our understanding of the dynamics of desert dunes by providing numerical chronologies where methods such as radiocarbon are frequently not applicable. Luminescence dating methods have allowed studies of the rates at which dunes form and move (e.g. Bristow et al. 2007), and the timing of periods of stabilisation of dunes (Leighton et al. 2014). Luminescence gives information about the timing of dune stabilisation, though whether such records indicate changes in aridity, sediment supply, or other factors (Kocurek 1998) is still a matter of debate (Thomas and Wiggs 2008; Chase 2009; Thomas and Burrough, this volume). As part of the INQUA Dune Atlas project (Lancaster et al. this volume), 385 luminescence ages (as of 23/11/2015) from the Arabian Peninsula have been compiled.

One of the challenges in compiling a database of ages is that luminescence methods have developed dramatically in the 34 years since Singhvi et al. (1982) working in the Thar Desert of India became the first to apply luminescence dating to desert dunes. Luminescence dating methods rely upon the growth of the trapped charge population within mineral grains as a result of exposure to ionising radiation in the natural environment (Aitken 1985; Duller 2008). In the laboratory the concentration of trapped charge can be measured by stimulating the grains either by heat or light to generate luminescence (thermoluminescence (TL) or optically stimulated luminescence (OSL)). The magnitude of the luminescence signal obtained from irradiation during burial is compared in the laboratory with the magnitude of the luminescence signal arising from irradiation using a laboratory radioactive source of known strength. This sequence of measurements yields the equivalent dose (D_e), an estimate of the radiation dose the sample was exposed to since the last exposure of the sample to daylight. To calculate an age, it is also necessary to measure the radioactivity of the sample, and this is termed the dose rate (D_r). The age is calculated by dividing D_e by D_r.

$$Age\ (ka) = \frac{Equivalent\ Dose\ (D_e)}{Dose\ Rate\ (D_r)} \qquad \text{Eq. 1}$$

Although the basis of luminescence dating is the same today as it was in 1982, there have been major changes in both the method of stimulating a luminescence signal (thermoluminescence (TL), infrared stimulated luminescence (IRSL), post-infrared infrared stimulated luminescence (pIR-IRSL) or optically stimulated luminescence (OSL)), and the different methods of equivalent dose determination (multiple aliquot methods versus single aliquot methods). Thus there is not a single 'luminescence' method but rather a family of different methods that share some common features, but also have their own specific traits. This paper briefly explains the difference between these methods and the impact that these changes may have had upon the quality and reliability of the ages generated. The paper also explores the impact that saturation of these different luminescence signals has upon the accuracy and precision of the ages that are generated. Examples from the INQUA dunes atlas are taken primarily from the Arabian region. Here, as in many of the major deserts of the world, the record of dune activity is likely to be a very long one, beyond the range of current luminescence dating methods. Inevitably samples are collected that push the limits of current luminescence methods, and this paper aims to explore the potential challenges involved in the analysis of such old samples.

2. Methods of luminescence measurement

2.1 Thermoluminescence: TL

The pioneering work of Singhvi et al. (1982) was the first to use luminescence dating methods to determine the period of time since the mineral grains making up the desert dunes had been last exposed to daylight. Singhvi et al. (1982) measured the thermoluminescence (TL) signal from quartz separated from sediments collected from dunes in Rajasthan. The TL signal from quartz had first been used to date the timing of firing of pottery (Wintle 2008), where the event being dated was heating of the quartz grains. Firing is able to remove all of the TL signal below a specified temperature (typically about 400°C), but exposure of quartz grains to daylight is only able to remove part of the TL signal. To be useful for dating, the method that was developed had to estimate the amount of the TL signal that was not removed by exposure to daylight and remained at deposition – this is known as the residual signal. This residual signal was then subtracted from the measured signal to allow an age estimate to be obtained (Singhvi et al. 1982; Duller 1996). The prolonged exposure of mineral grains to daylight prior to deposition made desert dunes an ideal target for this newly developed method, but the need for residual subtraction made it difficult to date very young sediments.

2.2 Optically stimulated luminescence: OSL and IRSL

In 1985 Huntley and co-workers published details of an alternative method of obtaining a luminescence signal from mineral grains. Instead of heating the grains to obtain a TL signal, the grains were exposed to light in a restricted wavelength range (514 nm in their instrument), and the resulting optically stimulated luminescence (OSL) emission could be used to determine the D_e. This method had the advantage that it only measured the part of the luminescence signal that was most sensitive to daylight, and thus there was no need to remove a residual signal. For desert dunes this made it possible to generate ages covering very recent periods of time, providing high temporal resolution for events in the last few millennia (Thomas et al. 1997).

The Research Laboratory for Archaeology and the History of Art in Oxford obtained an argon-ion laser like that used by Huntley et al. (1985) and undertook a great deal of research on desert dunes (e.g. Stokes et al. 1997). However, the expense and complexity of these lasers meant that no other laboratories made significant use of them, severely limiting the adoption of this method. Hütt et al. (1988) provided an alternative when they showed that it was possible to stimulate an OSL signal from feldspars using infrared light (~880 nm). Light emitting diodes of the type used in domestic remote controls for televisions provided an affordable and easily controlled source of IR, and many laboratories either built their own instruments or purchased affordable commercial systems. The application of infrared stimulated luminescence (IRSL) methods was also boosted by the development of single aliquot methods of D_e determination (Duller 1991).

However, a major challenge for IRSL measurements of feldspars was the phenomenon of anomalous fading (Wintle 1973). This term describes the loss of trapped charge at a rate that is much faster than would be predicted from a simple interpretation of the physical characteristics of the crystal defects at which charge is trapped. If anomalous fading occurs then it will lead to a measurement of D_e that is smaller than would be expected, and hence an underestimate of the age. A recurring point of discussion through the 1990's and early 2000's was whether anomalous fading was a universal phenomenon affecting all types of feldspars (e.g. Huntley and Lamothe 2001), or whether there were some feldspars that were not affected. Some authors chose to attempt to measure the rate of anomalous fading and correct for it, whilst others reported that they did not observe any fading, and thus did not correct. In Arabia, the very long sequence from Oman reported by Preusser et al. (2002; records ARBL0027-98) was dated using IRSL measurements, and they reported that they could not observe any fading. Until recently there has been little standardisation in how measurements have been made to assess the degree to which a sample of feldspar is affected by anomalous fading. In recent years it has become commonplace to measure the extent of fading and characterise it by a g-value (Huntley and Lamothe 2001). g-values are quoted as a percentage figure and values in excess of ~1.5% per decade are sufficiently large that they require some form of correction (Buylaert et al. 2012). Unfortunately many earlier publications do not express their fading measurements in this way, making it difficult to assess whether fading was significant or not.

While infrared (IR) light emitting diodes are able to stimulate an IRSL signal from feldspars, quartz requires a different light source. The Ar-ion laser originally used by Huntley et al. (1985) emitted light at 514 nm. In the late 1990's green, and then blue, light emitting diodes became commercially available, and these were ideally suited for generating an OSL signal from quartz (Galloway 1992; Bøtter-Jensen et al. 1999). Commercial instruments based especially upon blue LEDs rapidly became standard in luminescence laboratories, and together with the development of single aliquot methods of D_e determination (see below), this became a very standard method of analysis for desert dunes. The current database is now dominated by luminescence ages measured on quartz grains using the OSL signal generated using blue light emitting diodes.

3. Methods of equivalent dose determination: multiple aliquot and single aliquot

Calculation of the equivalent dose (D_e) from a sample involves comparing the intensity of the luminescence signal resulting from trapped charge acquired during burial of the sediment with the luminescence resulting from exposure of the sample to laboratory irradiations from radiation sources that are precisely calibrated (Duller 2008; Rhodes 2011).

Early luminescence work used many different sub-samples of each sample to determine a single value of D_e and these are termed multiple aliquot methods. Each sub-sample, called an aliquot, was given a different treatment: some were used to measure the luminescence arising from irradiation in nature, while others were used to measure the response to laboratory irradiation, or the response to exposure to daylight. All of these measurements on multiple aliquots were then combined together to generate a single value of D_e.

Duller (1991) described the first practicable method for making all of the measurements necessary for D_e determination on a single sub-sample. Such single aliquot methods are now the norm in luminescence dating. The methods proposed by Duller (1991) were designed for the IRSL signal from feldspars. Whilst these were used extensively through the 1990's (e.g. ARBL00245-251) difficulties were encountered when they were transferred for use with the OSL signal from quartz (Stokes et al. 2000). Murray and Wintle (2000) proposed a different single aliquot method termed the single aliquot regenerative dose (SAR) method. This method has proved extremely successful and has been tested extensively using samples with independent age control (e.g. Murray and Olley 2002 updated in Rittenour 2008). The SAR method, with blue LEDs as a convenient method of stimulating an OSL signal, has proven highly successful, as shown by the high proportion of ages in the Arabian database (76% of 385 ages) that have used this combination.

4. The challenge of obtaining reliable luminescence chronologies for older desert dunes

Farrant et al. (2015) have reviewed the database of luminescence ages for the northern Rub' al-Khali with the UAE, whilst Leighton et al. (2014) compiled a database focussing on the late Pleistocene, and the INQUA Dune Atlas builds upon these databases. The geographical spread of ages in the Arabian part of the INQUA dunes atlas (Fig. 1) shows that the majority of work done in the region has either occurred in the UAE or Oman, and the UAE ages are 67% of the total (258 out of 385 ages). Whilst Farrant et al. (2015) and Leighton et al. (2014) were able to see tightly clustered groups of ages in the Holocene and to a lesser extent within Marine Isotope Stages (MIS) 3-4, the luminescence ages demonstrate that a much longer record of aeolian activity is present, but it becomes increasingly more difficult to define as one goes further back in time. The reasons for this are discussed below.

4.1. Uncertainty on individual luminescence ages

The uncertainty on individual luminescence ages are typically in the range 5-10% of the calculated age. Fig. 2 shows for the suite of luminescence ages from Arabia that the majority of the younger ages (less than 30 ka) the uncertainty is typically 5%, especially for the quartz OSL ages generated using the SAR procedure. A small number of ages generated in the 1990's using a single aliquot additive dose method for the IRSL signal from feldspars have uncertainties that are significantly higher, up to 20%, though it is not clear how such large errors were determined.

The uncertainty in luminescence ages derives from the uncertainty in the values of both D_e and D_r used to calculate the age. In many environments the main source of uncertainty in D_r arises from uncertainty in the water content of sediments during burial. For ages based upon measurements of sand sized quartz grains a 1% change in the water content (expressed as weight of water divided by the weight of dry sediment) will typically produce a 1% change in D_r, and hence a 1% change in age. However, most desert dunes have very low water contents, and this source of uncertainty plays a relatively small role in the overall uncertainty on the age.

The other source of uncertainty arises from measurement of the D_e. When using multiple aliquot methods of D_e determination it is normal practice to only generate a single estimate of D_e, but with single aliquot methods it is common to determine D_e for 10, 20 or more aliquots. This larger number of measurements allows the uncertainty on the average D_e to be reduced significantly. In Fig. 2 uncertainties for ages calculated using multiple aliquot methods are typically nearer 10%, whilst those from single aliquot methods are nearer 5%.

A notable feature of Fig. 2 is that for samples whose age is greater than ~80 ka the uncertainty on the individual ages measured using any method are typically 10%, and often greater than this. This increased uncertainty derives in part from saturation of the luminescence signal, leading to greater uncertainty in the calculation of D_e.

4.2. Saturation of the luminescence signal

A key element in luminescence dating is measuring the growth of the luminescence signal in response to laboratory irradiation and determining the laboratory radiation dose that yields the same luminescence intensity as the signal resulting from irradiation during burial. This is the equivalent dose (D_e). The general shape of all dose response curves are similar and can be described by a saturating exponential equation of the form shown below (Eq. 2). A typical dose response curve for the OSL signal from a sample of quartz is shown in Fig. 3.

$$L_D = L_{Max}\left(1 - e^{\left(\frac{D}{D_0}\right)}\right) \qquad \text{Eq. 2}$$

where the luminescence intensity at dose D (L_D) is a function of the maximum luminescence intensity (L_{Max}), the radiation dose (D) and D_0, which is a parameter that describes how rapidly the signal reaches saturation. As part of the process of obtaining a value of D_e, in order to generate an age, laboratory measurements are made of the increase in the luminescence signal as a function of different doses. Where the luminescence signal increases rapidly with increasing radiation dose (i.e. at low radiation doses), it is relatively straightforward to use these measurements to determine the D_e for each aliquot. However, at high doses, the rate at which the luminescence signal changes becomes smaller, and it becomes increasingly difficult to use these measurements to determine D_e. Knowing how high a radiation dose can be reliably determined is difficult (Murray et al. 2002). There are potentially two complications and these are discussed below.

The first effect is a loss of precision as one approaches saturation. As with all measurements of physical quantities, luminescence measurements have uncertainties associated with them, arising primarily from the reproducibility of the instrumentation and the counting statistics of the luminescence signal that is observed. These uncertainties are propagated through into the final D_e (Duller 2007) and ultimately the age. As samples approach saturation, even though the uncertainty in the luminescence measurements remains proportional, the impact of these uncertainties upon the uncertainty in the D_e becomes much larger, and the uncertainties on the D_e become asymmetric (**Error! Reference source not found.**).

The second complication is the potential for a loss of accuracy due to systematic errors which may exist in the methods. The occurrence of this type of effect is much more difficult to assess, but Chapot et al. (2012) have made measurements on samples from Luochuan on the Chinese Loess Plateau where they were able to use the stratigraphic framework provided by the loess-palaeosol sequence to correlate to the MIS timescale, and thus provide independent age estimates for each of their samples. They observe a systematic mismatch between the luminescence signals that they observe in nature and in the laboratory which result in increasingly inaccurate age estimates. Chapot et al. (2012) observed a systematic underestimation of the real age of a sample as they approached saturation. However, since the cause of this systematic mismatch is unknown, it is unknown whether this is a general phenomenon that affects all quartz OSL measurements, and whether it is possible that some samples may exhibit systematic overestimation of age instead of underestimation.

The combined impact of these two complications upon datasets such as the INQUA Dunes Atlas is difficult to model because of the variability in the properties of the minerals being analysed (e.g. their D_0 values, Duller 2012), differences in dose rate, and the different analytical protocols that have been used. A major challenge in the study of aeolian activity in desert environments is that opportunities for obtaining samples from long sequences of stacked sediments are rare. Commonly samples are collected from relatively shallow depths (a few metres) because of limitations of access, and comparisons are made across large geographical distances. The Arabian dataset is unusual in containing a number of deep core records (e.g. Preusser et al. (2002) ARBL00027-98; Bray and Stokes (2003) ARBL00001-12; Stokes and Bray (2005) ARBL00130-142). Where these long sequences are available the impacts of the decrease in precision can be clearly seen, though it is not possible to judge whether there is any systematic deviation such as that seen by Chapot et al. (2012).

Wintle and Murray (2006) suggested that where D_e was beyond two times the value of D_0 (Eq. 2) the rate at which the luminescence was growing was very slow and so the results should be treated with caution. A simulation of the impact of saturation is shown in **Error! Reference source not found.**. The simulated dose response curve has a D_0 value of 50 Gy. Simulations were undertaken to show the variability in the D_e values that would be expected when working with samples in different parts of the dose response curve. At doses of 50 Gy or less (i.e. S1 on Fig. 4 which equates to a value of D_e that is equal to D_0) the growth of the luminescence signal with dose is relatively rapid and there is limited scatter in the resulting D_e values. However, when working near saturation (e.g. S3 on **Error! Reference source not found.** which equates to a value of De that is three times D_0) stochastic variation in the luminescence measurements results in very large scatter in the resulting D_e values. Differentiating between different episodes of dune activity would become increasingly difficult as the precision of the luminescence ages decreases. An increasing awareness of the challenge of working at high doses means that recent studies tend to give values of D_0 so that values of D_e can be compared with this (e.g. Farrant et al. 2015).

It is important to note that the limitation due to saturation is a limit in the size of the equivalent dose. Chapot et al. (2012) observed that the deviation between the expected and the calculated ages became significant above about 150 Gy. What this represents in terms of age depends upon the dose rate. If the environmental dose rate is low (e.g. 0.5 Gy/ka) then this would imply that it was possible to date reliably to 300 ka, but if the dose rate was 1.5 Gy/ka then the limit would be 100 ka.

4.3. New signals from quartz and feldspar: TT-OSL and post-IR IRSL

In the last decade two new luminescence signals have been explored in the search for signals that are able to overcome the problems of saturation of the quartz OSL signal and the problem of anomalous fading of the IRSL signal from feldspars, and these are described here.

The use of the SAR method applied to the OSL signal from quartz has been extremely successful in the last 15 years. However, one of the challenges with this method is that the OSL signal becomes saturated after irradiation with between 100 and 500 Gy of radiation (Fig. 3). This limits the time period over which dates can be obtained, and typically restricts analysis to the last ~150 ka. Wang et al. (2006) were the first to describe the use of a different luminescence signal from quartz called the thermally transferred optically stimulated luminescence (TT-OSL) signal which has the potential to date much older sediments. Exposure of a quartz sample to stimulating light from blue LEDs for between 100 and 200 seconds generates the OSL signal normally used in OSL dating. After this measurement the remaining OSL signal is near instrumental background. If the aliquot is then heated to 260°C and then cooled back to 125°C and blue LEDs are used for stimulation again, a new signal appears. This OSL signal arises from charge that has been moved from one defect to another by heating to 260°C, and hence the signal is termed thermally transferred OSL (see review by Duller and Wintle 2012). What makes this signal of interest is that whilst the OSL signal may increase for doses up to between 100 and 500 Gy and then cease to grow, the TT-OSL signal grows at doses up to many thousands of Gray (Fig. 3). Rosenberg et al. (2013) used a combination of OSL and TT-OSL applied to quartz from dunes buried by lacustrine sediments in the Nafud Desert and were able to obtain ages as old as 688 ± 71 ka (ARBL00300).

A new luminescence signal has also been explored from feldspars. The work of Thomsen et al. (2008), elaborated by Buylaert et al. (2012), described a luminescence signal from feldspars that appears not to be affected by anomalous fading. To obtain this signal, the IRSL signal that was previously used for dating is measured, but then the sample is heated to a high temperature (typically between 225 and 290°C) and whilst being held at that temperature the IRSL signal is measured again. This second signal, known as the post-IR IRSL signal, appears to be much less affected by anomalous fading. The dose response curve for this signal appears to be similar to that for the IRSL signal (Buylaert et al. 2009), and generally can be used to produce ages over a wider range than the OSL signal from quartz, but not as far back as the TT-OSL signal (Fig. 3).

One drawback to both the TT-OSL signal and the post-IR IRSL signal is that they are not reset by exposure to daylight as rapidly as the OSL or IRSL signals (Duller and Wintle 2012; Colarossi et al. 2015), and their resetting more closely resembles the behaviour of the TL signals from these two minerals. Thus TT-OSL and post-IR IRSL may not be well suited to dating recent events, but could be extremely valuable for dating older sediments.

5. Conclusions

Luminescence methods are ideally suited to dating deposition of desert dunes and have provided key data that has improved our understanding of their response to environmental change in the late Quaternary. The nature of luminescence methods is such that the majority of ages have uncertainties of between 5 and 10%, and currently there is no prospect of dramatically reducing these. The impact of these uncertainties is to make it increasingly difficult to resolve episodes of environmental change as one goes back in time. For instance, at 100 ka a typical uncertainty of 10 ka is almost half a precessional cycle. OSL from quartz is the signal that is most commonly used in luminescence dating, and for this signal the effect of saturation may also increase the problem for ages of 100 ka or older, leading to even worse precision. One potential approach to improve precision would be to average dates from multiple samples, and this could be a strategy for obtaining higher precision ages for key events. However, systematic deviations such as that seen by Chapot et al. (2012) which would result in increasingly inaccurate ages (though not necessarily imprecise ages) would not be resolved by making replicate measurements.

The last decade has seen the development of an increasingly diverse range of luminescence dating methods based upon measurement of different signals from both quartz and feldspar. Because these different signals grow at different rates (Fig. 3) and use different defects within the minerals, obtaining ages from each sample using these multiple chronometers has enormous potential for improving the reliability of older ages, and for extending the range over which luminescence dating can be used. However, there are clearly very large resource implications of obtaining multiple ages using multiple signals. For younger samples any increase in confidence in the age would be small and the increased effort would normally not be worthwhile. In contrast, for older samples, especially those approaching, or beyond, the range of the quartz OSL signal, the increased confidence in the age may well be sufficient to justify the increased resources.

In the INQUA Dunes Atlas Arabian dataset 122 of the 385 ages are beyond 100 ka. Almost half of these are from IRSL measurements of feldspars, but 55 (almost 15%) are based on measurements of quartz OSL. For the global database 7% of the ages are over 100 ka, equating to 265 ages. Thus there is already a significant database of old luminescence ages. Many of these were determined using quartz OSL but depending upon the dose rate these may face problems of saturation, and are likely to have large uncertainties. The development of luminescence methods based on new signals from quartz (e.g. TT-OSL) and feldspar (e.g. pIR IRSL) provide enormous potential in the future for obtaining more accurate ages over longer periods of time to explore the response of desert dune systems under a wider range of boundary conditions.

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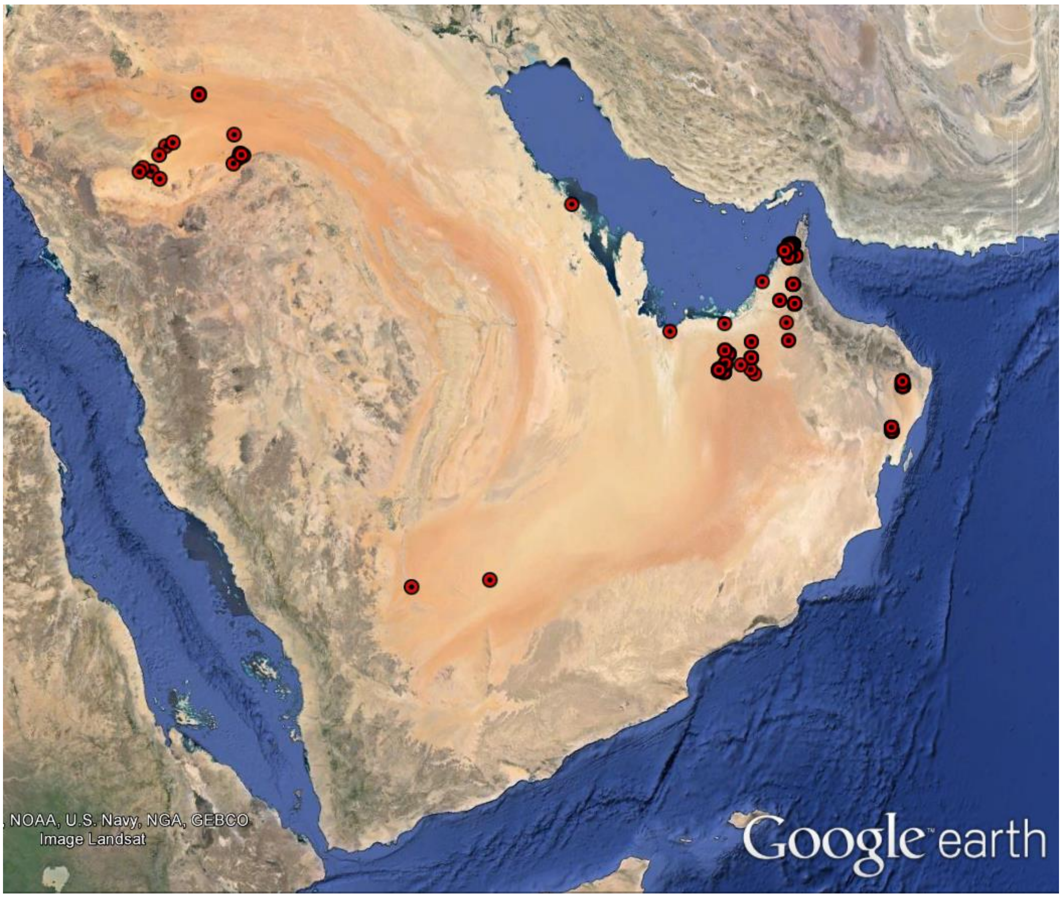

Fig. 1: Google Earth map of INQUA dune database coverage in Arabia showing concentration of effort in UAE and Oman, and also showing recent research in Saudi Arabia. Each red dot marks the position of one or more luminescence ages.

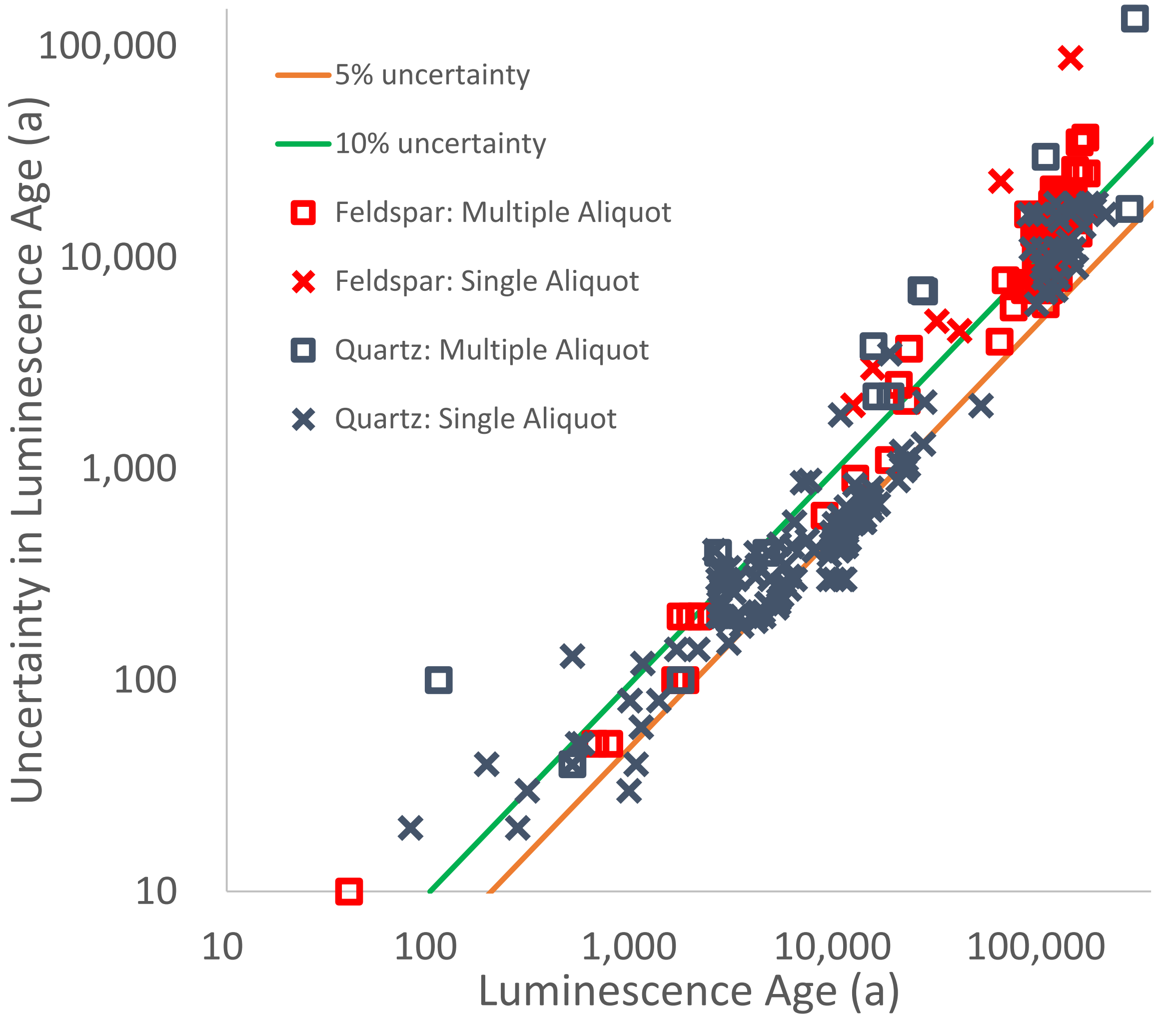

Fig. 2: Relationship between age and uncertainty for luminescence ages in the INQUA database.

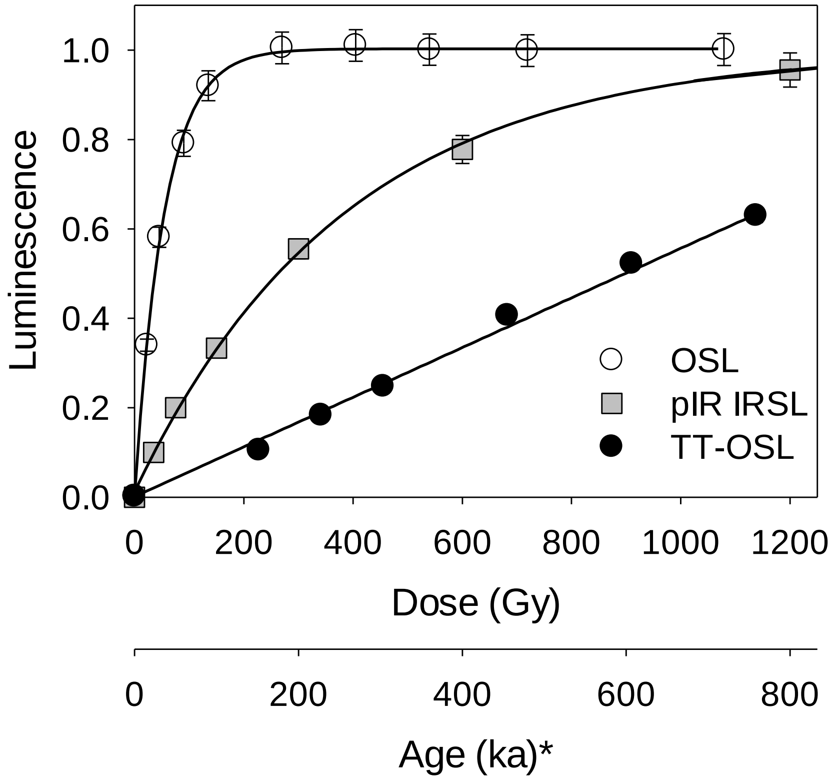

Fig. 3: Comparison of dose response curves from the OSL signal from quartz, the pIR IRSL$_{225}$ signal from feldspars and TT-OSL signal from quartz (data from Duller et al. 2015 and Buylaert et al. 2009). As well as plotting data as a function of dose, the equivalent age is also plotted on the x-axis assuming a dose rate of 1.5 Gy/ka.

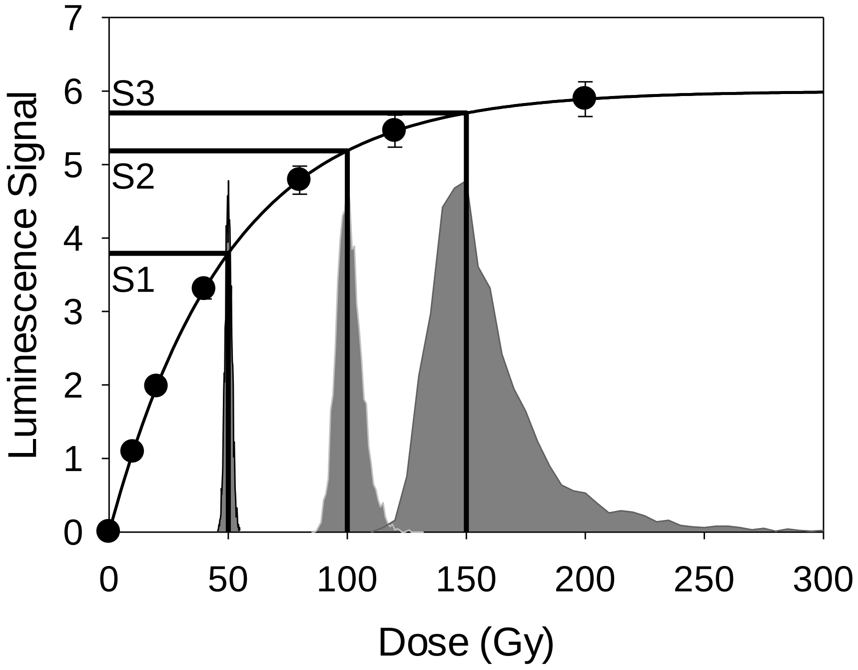

Fig. 4: The impact of saturation upon the variability in D_e, and hence age, for samples. Three different luminescence signal levels were simulated (S1, S2 and S3) such that they generate D_e values that are equal to D_0 (S1), to two times D_0 (S2) and to three times D_0 (S3). For each signal level 4000 D_e values have been simulated using a Monte Carlo process and the distribution of these D_e values are shown by the shaded areas. At three times D_0 (S3) the scatter in D_e values becomes very large, and becomes asymmetric because of the shape of the dose response curve.