Bateman *et al.* (2015) as published in Quaternary Geochronology, 30, 141-146. http://dx.doi.org/10.1016/j.quageo.2014.12.007

Instant Luminescence Chronologies? High resolution luminescence profiles using a portable luminescence reader

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Abstract

Establishing a robust chronology is fundamental to most palaeoenvironmental studies. However, the number and positioning of dated points is critical. Using a portable luminescence reader, it is possible to rapidly generate high resolution down core relative age profiles. Profiles of portable luminescence data from two coastal dunes were evaluated and compared with the results of particle size analysis, stratigraphy, and an independent historical chronology. Results show that, even in young samples, portable luminescence data is dominated by an age related signal which in homogeneous sediment need not be corrected for moisture, feldspar content changes or grain size. Profiles therefore provide relative chronologies from which accumulation phases can be established, and from which better targeted sampling and comparison to other sites could be undertaken. Even though they do not provide instant absolute chronologies, field-based portable luminescence profiling of Late Quaternary sites hold much potential to improve the resultant chronologies.

Keywords: dunes, particle size, portable OSL, Norfolk, Storms

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1. Introduction

The time dimension is fundamental to most Quaternary studies. Without a means of determining a chronological sequence, timing and duration of events, hiatuses within sequences, and correlation between sites is difficult or impossible to interpret. Most, if not all chronological techniques applicable to the Quaternary period rely on point sampling of appropriate material for a given technique. Yet, as has been shown, the location and number of samples taken from a stratigraphic sequence can have a profound impact on the resultant chronology. For example, Blaauw (2010, Figure 5), in his review of age-modelling, showed how very different age-depth models could be constructed depending on how ages were interpolated between actual dated points. Likewise, Telfer *et al.* (2010), in the context of regional-scale dune systems, showed that very different interpretations of externally-forced events could be made, depending on the number of dated samples used. Even where good bedding structures, bounding surfaces or archaeological features have been preserved and are visible in a sediment profile, as demonstrated by Leighton *et al.* (2013) at Rub' al Khali, Saudi Arabia, the preserved features may not have temporal significance and may lead to inappropriate sample collection. Obtaining an instant chronology of a profile while still at a field site would eliminate many of these errors that occur in the sampling and analyses of Quaternary sediments. The availability of an immediate chronology would allow for an initial assessment of the importance of specific sampling sites relative to the scientific questions the research aims to address. It would also inform where samples for other proxy records should be taken, allow focussing and higher resolution sample of areas which prove to be critical, and depending on the chronology developed, raise further questions about a site which could be addressed in the field.

Although vast improvements have been made to the methodologies of absolute dating (i.e. radiocarbon dating, luminescence dating, dendrochronology, etc.), and absolute chronologies can be available in as little as two days in the case of radiocarbon, these methods all require laboratory-based protocols that are not available during field sampling. Significant technological and methodological advances have also been made with luminescence dating over the last 30 years. There has been a shift in methodology from the slower to reset thermoluminescence signal, to faster optical stimulated luminescence (OSL) signal. There has also been a move from multiple aliquots individually loaded into measurement machines to produce a single palaeodose (D_e; e.g. Bateman 1995) to automated machines and protocols capable of generating a D_e from a single aliquot or single grain (e.g. Bateman *et al.* 2010). However, rather than producing ages quicker, these advances have been mostly used to generate more sample data to better understand and improve data and age quality (e.g. Bateman *et al*. 2010). As a result, whilst in principle it is possible to generate an OSL age based on a single aliquot measurement of D_e and by determining the dose-rate, it is highly unlikely that it would be viewed as acceptably robust. Most OSL ages are based on measurements of at least 24 replicates to check reproducibility and take steps to avoid incorporation of any antecedent (pre burial) signal and the effects post-depositional disturbance (e.g. Bateman *et al.* 2003; 2007), all of which takes time to attain the required laboratory precision.

Until recently, instantly derived chronologies remained largely aspirational. In terms of luminescence, it is technically possible to undertake measurements of luminescence in the field. One such instrument that enables this is the Scottish Universities Environmental Research Centre (SUERC) portable OSL reader (Sanderson and Murphy 2010). This machine uses pulsed or continuous wave stimulation either by infrared light (IRSL; IR LEDs at 880nm with RG780 filters) or by blue light (OSL; Blue LEDs at 470nm with CG420 filters) with signals detected through UG11 filters by a photon counter (Sanderson and Murphy 2010). The availability of IR and OSL measurements allows for the potential to target either feldspar or quartz related signals. Each measurement takes 60 seconds (or other set control time), during which time the photon counter collects and calculates the IR or OSL signal and measurement. Actual age determinations are not possible with this method, as the instrument lacks a radiation source and heating system, both necessary to thermally assist OSL signals and to quantify sample specific sensitivities. Instead, the total luminescence count measurements can be viewed as a rough proxy for time, with older samples having a higher luminescence signal than younger samples.

Fundamental to the use of the portable OSL reader is that the measured signal must be dominated by a luminescence signal which relates to age since sediment burial. Luminescence signal is potentially a function of a wide range of variables, including mineral composition, particle size, colour, moisture content and age. Different minerals contribute not only luminescence in different wavelengths but also accumulate luminescence and are bleached at different rates; even within the same mineral, the ability of grains to accumulate dose (i.e. their sensitivity) can vary. The luminescence signal derived from a sample from any given location is generated by the background dose-rate, which is a function of not only uranium, thorium, and potassium levels, but also of whether pore spaces are filled with water or precipitated cements, which attenuate the ionizing radiation. Sediment size and depth from the surface also affects the cosmogenic dose. Thus when comparing multiple down profile measurements to establish a relative chronology, as for example presented by Munyigwa *et al*. (2012), it is necessary to know whether differences in the total luminescence signal do reflect age or these changes in sediment/luminescence characteristics.

This study aimed to generate portable OSL (POSL) profiles in order to test whether data changes reflect depositional events or other factors, and if the data reflects other factors, which factors other than age affected the luminescence signal. In order to avoid any substantial changes in sediment type, single dunes were selected. To rigorously test the approach, rather than apply it to ancient dunes with long durations and large temporal hiatuses, historical to modern dunes were sought. The availability of historical records was also required, so that these could be used as a known comparative chronology. To this end, the coastal dunes at Holkham, Norfolk fulfilled all of these criteria.

North Norfolk has a low-lying coast with a moderate to low wave regime from the north-east, a westerly longshore drift and macro-meso tidal ranges (Andrews *et al.* 2000). Its wide sandy beaches and/or subtidal sand flats have given rise to extensive coastal dunes that form multiple coast parallel barriers up to 10 m high. The coast at Holkham is known to have been prograding during the Holocene (Andrews *et al*. 2000). Historic maps (1st series ordnance survey) and aerial imagery (Crown copyright Royal Air Force aerial photographs 1946, Norfolk County Council aerial survey 1988 and Get Mapping imagery 2007 via Google Earth) show the coast to have continues to prograde and the back beach dunes are still actively accumulating sediment today. Two dunes were selected for this study. Site 1 is located at the back of the present beach, on dunes presumed to be the mostly recently formed; site 2 is located on a line of dunes parallel to the coast, approximately 300 m inland, and interpreted as an older formation than the dunes around site 1.

2. Methods

At both dune sites sampling was carried out on the dune crest using a Dormer engineering sand drill. This was used to core each dune from crest to dune base. At ~25 cm intervals, samples for POSL measurements were collected in light-tight chemical photographic film canisters (Fig. 2). Samples for full OSL dating were also collected from near the dune surface, mid core and base of core (Fig. 3). Light-contaminated sediment from the ends of the sample from these were also used to supplement the samples for POSL measurements and to provide direct comparison between the two measurement methods. This resulted in the collection from Site 1 and 2 of a total of 35 samples for POSL measurement and 6 for OSL dating. Results of the OSL dating form part of a wider study; this paper will only summarise the results of the quartz based single aliquot regeneration based OSL ages.

Whilst POSL measurements could have been conducted on site, to achieve more controlled measurements and better evaluate signal changes, samples were transported to the Sheffield University luminescence laboratory. In the dark room, the sediment from the top and bottom of the canister (potentially light contaminated during sampling) was removed and used to evaluate moisture content and for particle size analysis. The remaining sample material was dried at 30^{o}C for 24 hours before POSL measurement was conducted.
To factor out the POSL signal variability due to changing sample volume and changing geometry in respect to the portable reader, a small amount of each sample (~5 g) was placed as a monolayer across the entire base of a 5 cm diameter petri dish. This dish was then placed in the portable reader for measurement. Repeat measurements using this approach were able to produce good reproducibility in terms of both IR and post-IR OSL signal measurements (Fig. S1). Each sample underwent 60 s continuous wave stimulation with IR followed by 60 s continuous wave stimulation with blue light. Data in both cases was integrated 1 s bins. The initial IR measurement should have been collecting the luminescence dominated by feldspars, whilst the post IR OSL measurement should have targeted the quartz dominated signal. Repeat IR measurements of the same sample without removing it from the machine between measurements revealed that full feldspar depletion was not achieved within 60 s. This is in part due to the limiting of the IR stimulation power to only ~90mW in order to allow the portable reader to be powered by batteries (Sanderson and Murphy 2010). It also reflects the much larger sample size being measured. A standard 9 mm OSL aliquot typically measures approximately 1,800 grains of 200μm diameter simultaneously, whereas the portable reader

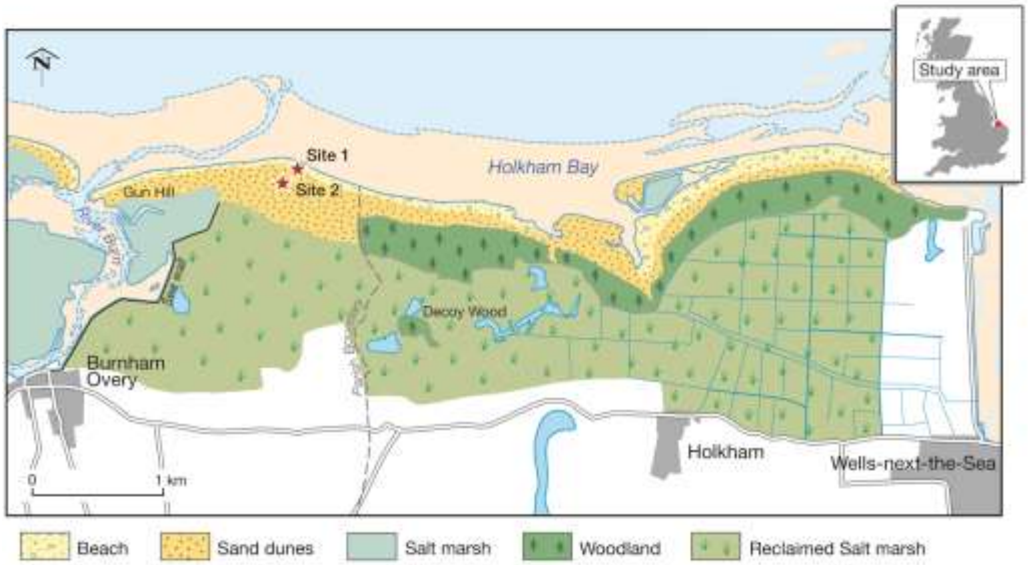

Figure 1: Sampling site at Holkham dunes. Site 1 from dunes at back of present beach, site 2 from a line of dunes inland from those associated with Site 1.

measures approximately 60,000 of the same diameter grains at a time. Whilst this limits the effects of grain heterogeneity and has the potential for measuring a signal multiple times from the same sample, it also results in much slower shine down of the luminescence signal. Due to these limitations, the post-IR OSL signal cannot be considered a pure quartz derived signal, as it will still contain a component of feldspar derived signal.

Given the sampled context for this study with sediment being directly derived from a single source (sub-tidal sandflat and associated beach) with a high probability for multiple bleaching-dosing cycles, likely changes in sensitivity down core were considered low. Use of photo-transfer induced by UV light has been successfully used elsewhere to account for down core sensitivity changes (e.g. Li and Wintle 1994) however the extended bleaching and UV exposure required for each sample (> 60 mins) is counter to the production of quick data for evaluative purposes so was not undertaken. Sensitivity was therefore measured on unprepared sediment samples in a Riso Da-18 with a calibrated $^{90}Sr/^{90}Y$ beta source and luminescence detected through a 7.5 mm thick Hoya U-340 filter. A modified single aliquot regeneration (SAR; Murray and Wintle, 2003) protocol was used with a preheat of 260°C for 10 s with OSL measurements made at 125°C for 60 s following a 60 s IR wash at room temperature. From this, the response to the standard test dose after the natural OSL had been measured was used to derive comparative sensitivity data. These measurements were also used to derive De values down each core, and subsequently compared to the POSL data.

Each POSL samples also underwent particle size analysis to check whether changes in grain size had an impact on the POSL signal as measured. Results are also used to indicate subtle changes in depositional environment as reflected in mean size and sorting. Particle size analysis was conducted using a Horiba LA-950 laser diffraction particle size distribution analyser. Samples did not require sieving, as grain sizes were all under 1.4mm, but all samples were split down to a suitable size using a riffle box. The reduced samples were suspended in 0.1% sodium hexametaphosphate, before dispersal in de-ionised water within the instrument using ultrasound and pumping. The laser diffraction technique calculates the mean and median grain size of each sample (Φ), as well as sorting, skewness, and probability distribution; this paper utilises the mean grain size as a representation of particle size. Moisture content was also analysed by recording pre- and post- drying weights of the sampled sediments. Moisture is presented as a total percentage.

The data from the above measurements were compared in various combinations in order to test whether burial age, sediment size, moisture or varying amounts of feldspar to quartz could account for changing post-IR OSL down the two sampled cores.

3. Results

Measurement of the background radioactivity at the two sites using a portable gamma-spectrometer showed the dose-rates of all samples were consistent within 2 sigma errors so could not be significantly affecting POSL results. Sample size, colour and the measurement geometry were also consistent across the study area (see above) and were therefore not evaluated further.

As shown in Figure 2a and 2e, the POSL increased with depth at both sites, as would be expected in a sedimentary environment conforming to the law of superposition. Particle size results showed all samples to be well to moderately well sorted, unimodal with mean values in line with those found for dunes elsewhere (e.g. Lancaster 2013). Site 1, closest to the present-day beach, overall has a lower POSL signal than Site 2 confirming the initial interpretation that it is a younger dune. At site 1 data are separated into two significantly different groups (at 95% confidence level); the uppermost group contains three points which have a mean signal of 680 ± 110 cts, and the remaining data group has a mean signal of 870 cts (Fig. 2a). Likewise, Site 2 data are separated into three groups (at 95% confidence level); the uppermost three points have a mean signal of 740 ± 160 cts, a middle group of four points has a mean of 970 ± 100 cts, and the remaining data

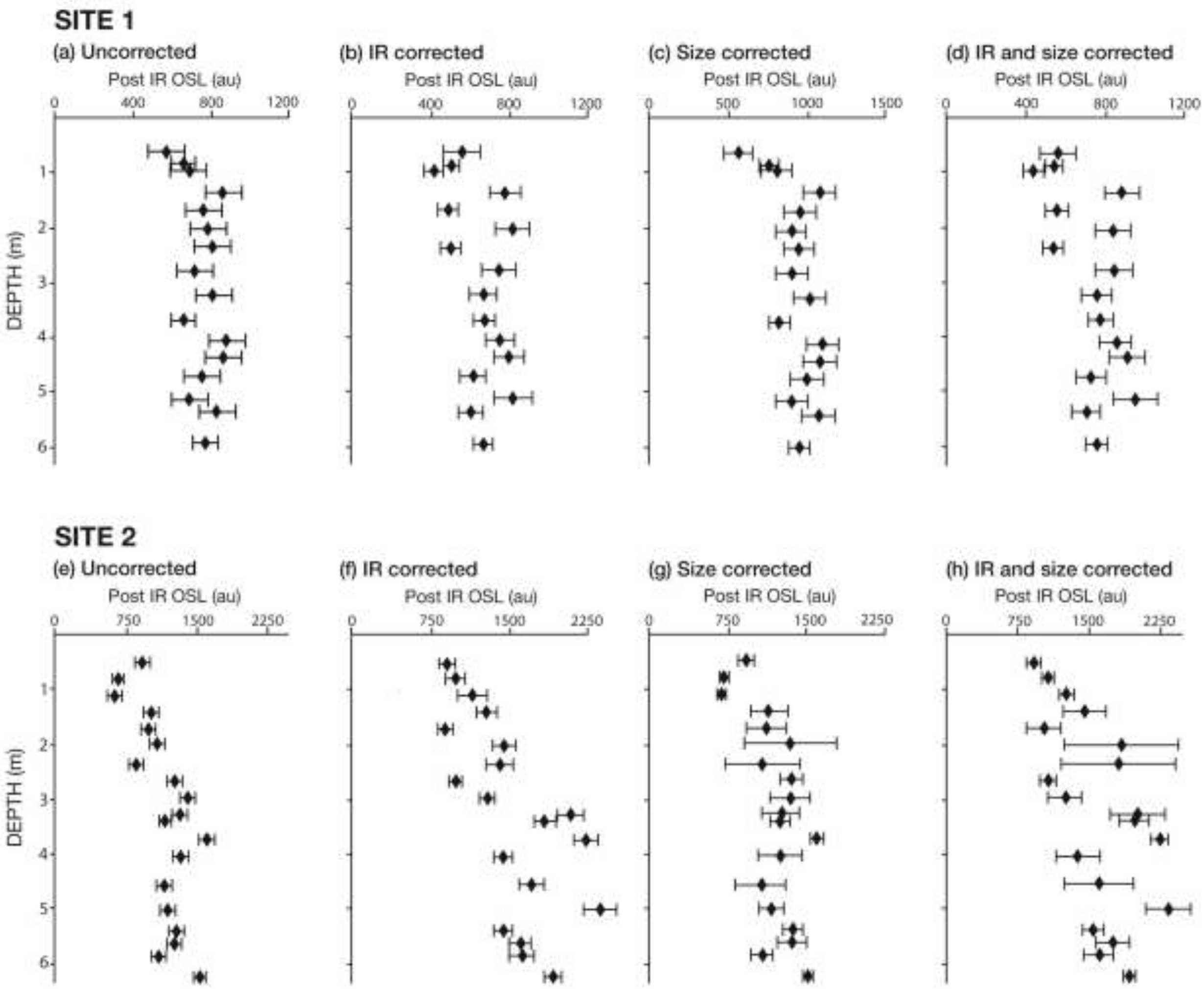

Figure 2: Down profile portable Post IR OSL results from sites 1 and 2 uncorrected and corrected based on IRSL and mean particle size measurements. Errors shown are based on

have a mean signal of 1290 ± 150 cts. Data from both sites show considerable down-core inter-sample variability.

Whether the above groupings reflect different phases of dune construction (i.e. age) or other factors was also examined. The post-IR OSL data was corrected by the IR/POSL ratio, by mean grain size, and by moisture, as both individual normalising factors and in combination with other factors (Fig. 2, Fig. S2). Variability of IR and mean grain size down-core at both sites was not substantial, but correcting for these factors separately (Figs. 2b, 2c, 2f, 2g) and in combination (Figs. 2d and 2h) had an apparent positive impact in better separating most of the groups identified above. There was one exception to this seemingly corrective combination: when data was corrected for both size and IR at site 2 the upper two groups became indistinguishable (Fig. 2h). However, correction with IR and size (both individually and in combination) also increased the inter-sample variability, and if these were the major (and only) controls on POSL signal, this would not be the case. Correction using moisture, either individually or in combination with the size and IR data, appeared to only increase inter-sample variability (Fig. S2).

To further check whether the groupings of the POSL data reflected changes in depositional age they were compared to site stratigraphy and the full quartz SAR ages (Fig. 3). For site 1, the grouping of POSL data into two, conforms with the logged upper unit which has a slightly younger SAR OSL age (albeit within errors) than the sediment below. These results show that down to 1 m is better sorted and coarser than the sediment below although it is noted that variability in particle size results at around 2 m (coarser and slightly poorer sorting) is not reflected in the POSL results. For Site 2, an upper unit is separated from an undifferentiated lower sand unit by a thin palaeosol at 1m depth but the lower third phase was not logged when coring. Particle size results show a very similar trend to the POSL data. The mean size and sorting of the upper most two samples and those from below 2.2 m are much finer and better sorted than samples between 1 - 2.2 m. This adds credence to the POSL tripartite sequence within this dune profile. The upper unit quartz SAR OSL age is much younger than that of the basal age indicating that two different age units definitely do exist. Unfortunately the mid-core quartz SAR OSL could only provide a maximum age. It was sampled without the benefit of the POSL down core data and appears to occur as a point in the profile where significant variability within both the POSL and size data is occurring. The reversal in POSL data at this point is replicated in the down-core De data (Fig. S3), in a coarser poorer sorted sand, and in sensitivity tests where a large sensitivity change occurs (Fig. S3)

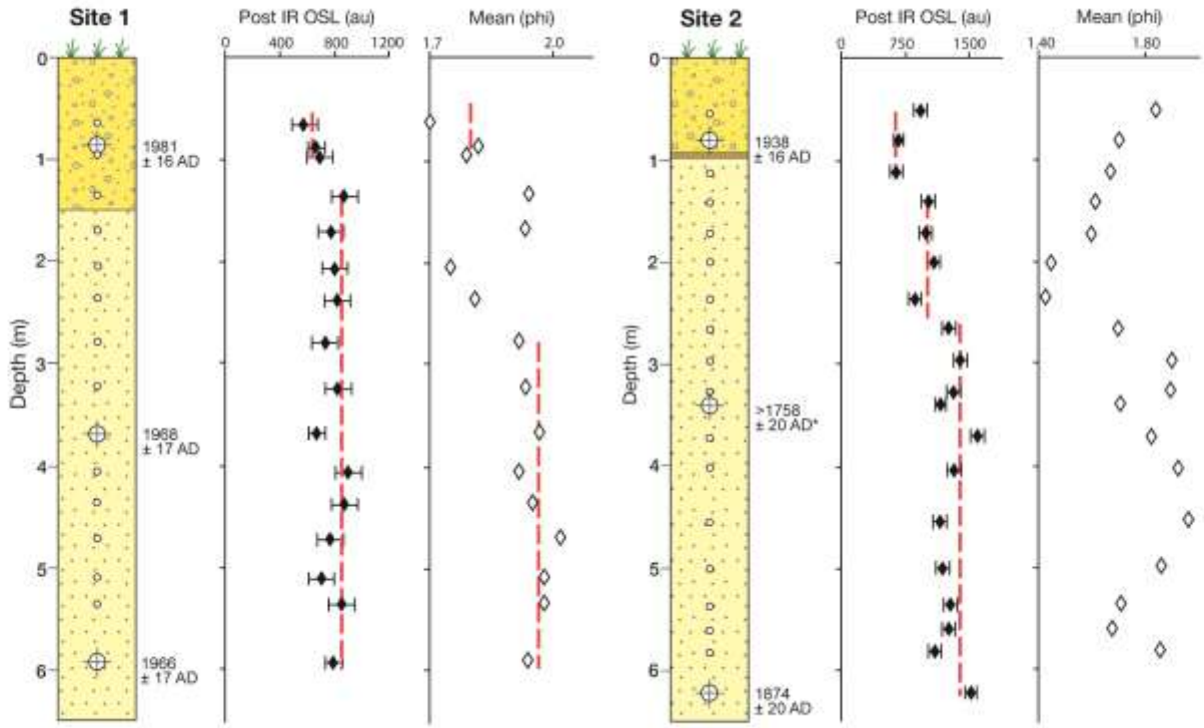

Figure 3 Stratigraphy of two dunes samples compared with down-core post-IR OSL portable measurements and mean particle size. Note dashed lines indicate observed groups of data.

Discussion

With regards to data correction, the fact that moisture correction was not successful may reflect the inherent nature of sampling, in that the moisture data was based on water content at the time of sampling, rather than estimates of average water content during burial (not always an accurate representation/guesstimate). As such, it holds the promise that had the measurements been carried out in the field, results and the interpretation of them would have been very similar. That other corrections made little difference probably reflects the fact that for this study only well to well-sorted fine to medium sand was measured which was all wind blown from the same beach. As shown elsewhere (e.g. Sanderson *et al.* 2003; Kinnaird *et al.* 2011; Muñoz-Salinas *et al.* 2011), where more varied sediment types are profiled then correction might be of more significance. Given the IR data are generated as part of the Post IR OSL then seeing whether this is the case does not detract from the principle of rapid on site profiling. Particle size cannot be carried out in the field at the time of sampling but as it is often carried out as part of wider site evaluations, corrections for it can be used for post-site visit evaluation of the data and may be of some value.

Based on supporting stratigraphical, SAR OSL and particle size data, for site 1 it appears that the two phases identified in the POSL data is reflecting small changes in age. At least two phases identified in the PSOL data are also confirmed by stratigraphy, particle size and age. Whether the group identified within the POSL data between 1.5 – 2.5 m at Site 2 truly reflects an age difference within the core cannot be firmly established. The latter is presumed to indicate an influx of differently sourced sediment at this phase of dune building. The fluctuation in POSL data is not caused by changes in size, IR or sensitivity (Figs. 2f and 2g; Fig. S3). If the significant coarsening of sand just below 2.5 m reflects dune modification during a storm event bringing differently sourced sediment, then sediment below this point would relate to an older phase of dune building. Such a link was posited by Clark and Rendell (2006) in the context of North Atlantic Dunes, where climate oscillations led to the deposition of sediment from another offshore location. In this case, whilst undiscernible within the stratigraphy, the lower unit would be split into two temporal units as per the POSL data. Results from down-core SAR D_e measurements show this to be the case (Fig. S3) and that three phases as per the POSL profile are probably present.

Historical maps and imagery show that neither site could have existed prior to 1840 as where they were found was part of the beach prior to this time. Coastal progradation after this date would have provided accommodation space for the formation of the dunes associated with Site 2. Maps show that Site 1 (presently at the back of the beach) only appears above the high-tide line after 1970 and first appears on an aerial photograph from 1988. Documentary evidence therefore supports both the full SAR OSL ages and also the differences in POSL found between the two sites.

Other field studies have shown increased POSL with depth (e.g. Munyigwa *et al*. 2012, Figures 6 and 7) and, as with this study, have shown it is possible to identify stratigraphic breaks which are not necessarily discernible (e.g. Kinnard *et al.* 2012; Muñoz-Salinas *et al.* 2011). POSL profiling of very recent sediments, such as investigated in this study, appears to be successful and supports the findings of Muñoz-Salinas *et al.* (2012) who successfully used PSOL data from deposits which were in the 10-500 year range. Some studies have used the inter-sample variability to indicate reworking (e.g. Burbidge *et al.* 2007) or changes in provenance (e.g. Muñoz-Salinas et al. 2012). Inter sample variability and how quickly the POSL signal depletes has also been used to indicate whether sediments are well or poorly bleached (e.g. Kinnaird *et al.* 2012; Muñoz-Salinas *et al.* 2012; Muñoz-Salinas *et al.* 2014). This current study shows that where samples are both recent and from a depositional context with poor bleaching,inter-sample variability can not be accounted for in this way. The persistence of inter-sample variability (even after corrections) does effect the precision to which temporal hiatuses might be identified to. It also realistically means that profiling is better suited to giving an overall indication of magnitude of sedimentary unit age, hiatuses between units and whether deposition was slow gradual accumulation or temporally staged. Despite this, the intensity of sampling possible in the field, ease of measurement and low cost in terms of measurement time and expense which can be achieved shows this approach to have much utility.

Conclusions

In summary this study has shown that:

- It is possible to rapidly generate accurate, high resolution down core OSL data using a portable OSL reader. Generation of both IR and post-IR OSL using the portable system took less than an hour per sampling site, and would do so irrespective of whether they are carried out in the field or laboratory.
- While they do not provide an instant chronology, down core POSL profiles do provide high resolution relative age profiles. The POSL data successfully differentiated the older dune from the younger one, and multiple phases of dune activity within both profiles.
- Although the sediments were young, and consequently the age related POSL signals were relatively weak, age signals appear to still have dominated the post-IR POSL measurements. IR and other data were generated to correct for non-age related variables which may have contributed to the POSL signal, but in the context of the fairly homogeneous sediments in this study, little was gained by doing these corrections. In contexts where there are a wider range of sediment types then correction for moisture, size and IR may become more significant. As IR data is generated at the same time, this is readily available to check.
- POSL down core measurements are capable of detecting recorded and observable temporal changes in sediment profiles, but they are also capable of detecting major changes and events that are not visible based only on changes in physical characteristics of sediments.
- POSL profiles could be used to better target sampling and for comparison to other sites. For example, retrospective consideration of the mid-core sample at site 2 should have been taken higher in the profile to resolve whether two or three phases of dune building had occurred at that site. This could have been solved and corrected in the field with the application of POSL measurements

As such we conclude that field-based use of portable OSL profiling of Late Quaternary sedimentary sections and cores holds much potential to improve the resultant chronologies associated with such sites, even if in themselves they do not provide instant chronologies.

Acknowledgements

Sarah Henderson and the Holkham Estate is thanked for their assistance in working at Holkham. Gill Tyson is thanks for her cartographic assistance with the figures. This research was funded through a White Rose Consortium Grant held by Dr Katherine Selby at the University of York.

Supplementary data

Supplementary data related to this article can be found at http:// dx.doi.org/10.1016/j.quageo.2014.12.007.

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