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ScienceDirect
Quaternary Geochronology 2 (2007) 296–302
QUATERNARY GEOCHRONOLOGY
www.elsevier.com/locate/quageo

Research paper

Survey of Palaeolithic sites by luminescence profiling, a case study from Eastern Europe

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Received 12 October 2005; accepted 1 May 2006
Available online 11 July 2006

Abstract

A luminescence-based approach to the rapid characterisation of sites in terms of their datability and sedimentary history is described (luminescence profiling), and contrasting results presented for three Palaeolithic archaeological sites in Russia. Three mineral/grain-size fractions (polymineral silt-sized, polymineral sand-sized, quartz-enriched sand-sized) were separated from each of many small samples taken through the sites' stratigraphies. Luminescence signals from the different fractions were measured using infra-red light, blue light, and thermal stimulation. Patterns in luminescence sensitivity and equivalent dose values are used to indicate which sites, samples, fractions and/or signals may yield meaningful luminescence ages, and the experimental effort that may be required to obtain said ages. Case studies range from a site where a detailed chronostratigraphy might be produced using a limited number of full luminescence dates to tie in the profiling results, to a site where profiling indicates there is little chance of producing useful luminescence dates. These interpretations are based on dialogue between the luminescence profiling results and archaeological, palaeoenvironmental, and geomorphological evidence. Luminescence profiling is shown to be a useful tool for understanding sedimentological process at each site, independent of full luminescence dating.

Keywords: Luminescence; Sediment; Palaeolithic archaeology; Palaeoenvironment; Russia

1. Introduction

Luminescence dating forms an integral part of the project, ''Neanderthal climate preferences and tolerances: the need for a better chronology'', part of the Natural Environment Research Council (NERC) thematic programme, ''Environmental Factors in the Chronology of Human Evolution and Dispersal'' (EFCHED). The overall aim of the project is to investigate whether present chronological data are biasing our perception of the relative cold adaptation of Neanderthals and modern humans, focussing on a region of continental climate northeast of the Black Sea (Fig. 1; Housley et al., 2006). The aims of the luminescence-dating programme are to provide age estimates, and broader chronostratigraphic context, for key archaeological and palaeoenvironmental evidence from a variety of Palaeolithic sites in the region. This programme is focussing on sediments, which are present in all the contexts of interest but generally appear challenging for luminescence dating. To aid the selection of sites and layers upon which to focus, many small ''profiling'' samples were taken through the sections at the same time as the full dating samples.

From initial prioritisation of the 15 sites sampled in the 2004 field season (Fig. 1; Burbidge et al., 2005), seven were selected for luminescence profiling. The profiling samples were subjected to simple, rapid preparation and luminescence measurement, to provide indicative values of sensitivity and absorbed dose through the stratigraphies. This paper describes the laboratory procedures used, and demonstrates the effectiveness of luminescence profiling for assessing sample behaviour and stratigraphic variation, using contrasting examples from three of the sites.

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1871-1014/$ - see front matter © 2006 Elsevier Ltd. All rights reserved.
doi:10.1016/j.quageo.2006.05.024

Fig. 1. The region northeast of the Black Sea, showing the locations of all sites sampled in the 2004 field season.

2. Luminescence profiling

Luminescence profiling aims to rapidly produce a stratigraphically detailed survey of a site or series of sites. Its objectives are to assess the presence and suitability of particular minerals/grain-sizes/signals for full luminescence dating measurements, and to provide a record of variations in luminescence and related characteristics that can be integrated with archaeological and sedimentological interpretations.

Luminescence profiling utilises simple measurement techniques akin to those normally applied in exploratory luminescence measurements (e.g. Hamel and Huntley, 2003), or those applied to large numbers of subsamples for the assessment of D_e distributions (e.g. Olley et al., 1999; Spencer et al., 2003): profiling measures the spatial distribution of results across a site or series of sites. infrared light, blue light, and thermally stimulated luminescence (IRSL, OSL, and TL) are measured from the same subsamples in order to compare the different luminescence responses. This approach has previously been applied to sand-sized quartz and polymineral fractions in dating studies conducted at SUERC (e.g. Sanderson et al., 2001, 2003; Bicket, 2004), and in the present study it has been extended to include polymineral silt-sized grains.

The utility of making separate luminescence profiling measurements requires that it not only provides information relevant to the mineral or grain-size fractions likely to be used in full luminescence dating measurements, but that this information is obtained rapidly with relatively little effort. To this end, a basic series of preparatory treatments has been used, to separate fine (4–11 μm) and coarse (90–250 μm) polymineral fractions, and a coarse HF etched fraction expected to be dominated by quartz (Fig. 2).

Approximately 5 g of bulk sample material was first wet sieved, to reduce the chance of contamination by geological grains from limestone clasts. Carbonates were then removed from the 90–250 μm fraction using HCl, and after thorough rinsing in water to clean the grains, sufficient of the polymineral coarse material was removed to make up three aliquots. Remaining material was HF etched to produce a quartz-rich fraction. The less than 90 μm fraction was settled in water for 2 min and the suspended (less than ca. 20 μm) fraction collected and centrifuged out for further processing. Approximately 2 ml of this was treated in H_2O_2 and HCl to remove organics and carbonates, and the undissolved material settled in acetone to isolate the 4–11 μm fraction, which was itself settled onto steel discs in acetone. It was found convenient to process 16 samples in parallel up to this point in one working day, the coarse grain discs being prepared the following morning.

Two aliquots from each fraction were subjected to simple regenerative D_e determinations, using IRSL, post IR OSL, and post IR & OSL TL for the polymineral fractions, and sensitivity corrected OSL for the HF etched fraction (Table S1). This rapidly produced large matrices of

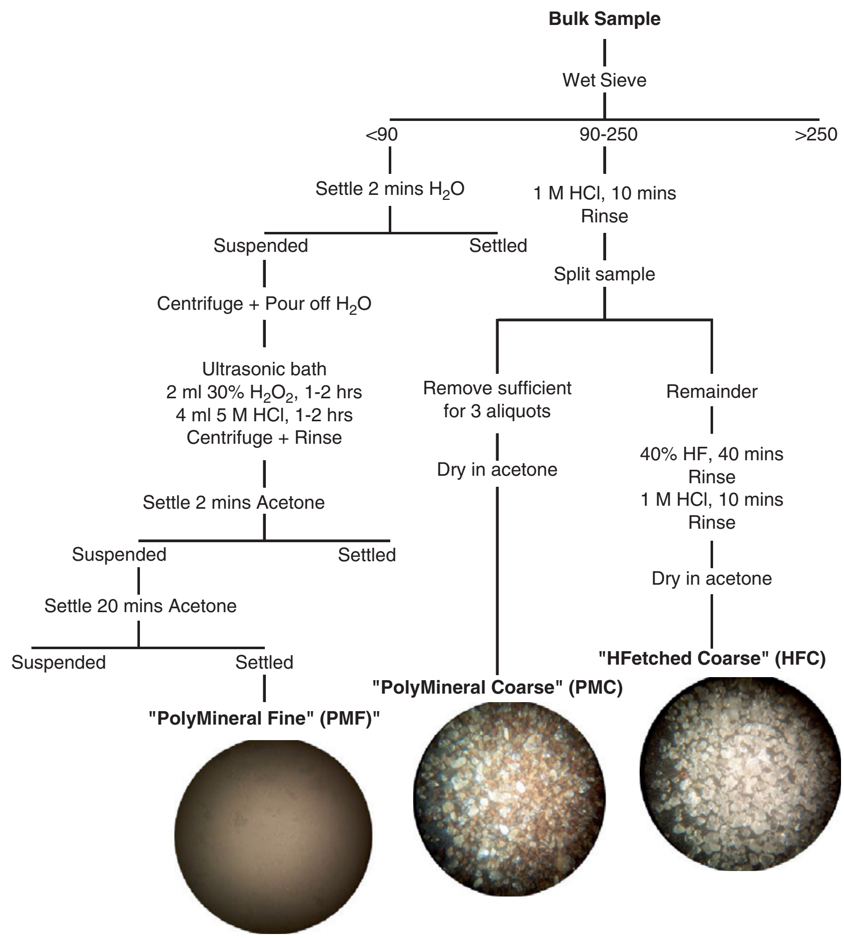

Fig. 2. Preparation of "polymineral fine", "polymineral coarse", and "hydrofluoric etched coarse" mineral/grain-size fractions from profiling samples.

luminescence sensitivity and D_e values through the sections sampled, including paired reproducibility assessment (e.g. Table S2, Fig. 3).

3. Results

D_e values are plotted in stratigraphic order in Fig. 3, along with results of field γ spectrometry to indicate changes in dose rate down the sections. Guidelines link the plots to certain stratigraphic features in the adjacent section diagrams. No strong relationships were observed between sensitivity and stratigraphy within the sites, but the average values for each mineral/grain-size fraction did vary substantially (Table S2).

3.1. Kostienki 14—Markina Gora (Sinitsyn, 2003)

3.1.1. Background

A silty colluvial site by the River Don near Voronezh, Kostienki 14 is one of a number of sites in the area, with long histories of excavation. This site had relatively good independent age control on Early Upper Palaeolithic archaeological levels, sampled from two overlapping sections. For simplicity the layers have been numbered 1–11 in Fig. 3(a), instead of following the Kostienki system. The upper part (layer 1–2) included a tephra layer that has been associated with the "Campanian Ignimbrite/Y5", constrained to ca. 39.3 ka by ^{40}Ar/^{39}Ar dating and δ^{18}O stratigraphy (Pyle et al., 2006). Layer 6 is a palaeosol containing evidence for a magnetic excursion, and is thought to have been deposited ca. 40–42 ka or ca. 44–46 ka ago (Sinitsyn, pers. commun.; Sinitsyn, 2003). ^{14}C results in association with the tephra and palaeosol are around 32 and 36 uncal. ka BP, respectively. Kostienki 14 was considered a priori to be relatively amenable to luminescence dating, since IRSL ages of 34.3 ± 2.9 ka and 44.9 ± 3.8 ka (UIC-749, UIC-748, Sinitsyn, 2003) had already been obtained from levels stratigraphically equivalent to layer 10 (Fig. 3(a)).

3.1.2. Questions

The 2.5 m of section sampled was expected to have accumulated through colluvial and related mechanisms during ca. 5–10 ka, but different phases of accumulation were apparent in the strata. So, in addition to the general points outlined in Section 2, the particular questions to be addressed by profiling this site were: to what degree were luminescence signals reset (at deposition or by reworking)?

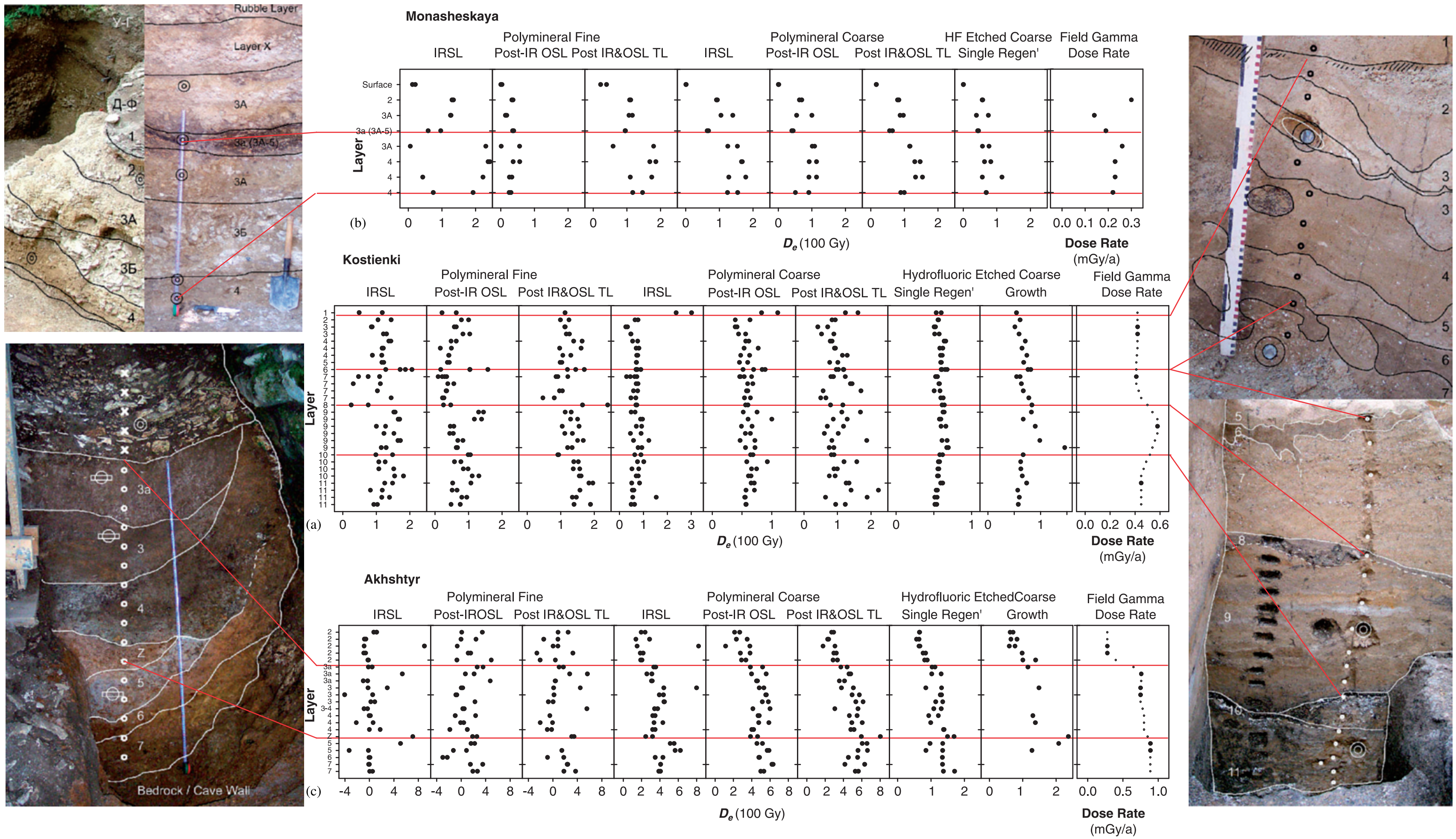

Fig. 3. Luminescence profiling results from three contrasting sites, with field γ-spectrometry results and section diagrams. Samples are plotted in stratigraphic order rather than by depth, since more than one section was sampled at two of the sites. Note changes in D_e scale with mineral/grain-size fraction. Smaller points in plots of field γ-dose rate are interpolated values. In section diagrams, small circles or crosses indicate locations of profiling samples, larger circles indicate locations of full dating samples and γ-spectrometry measurements.

and: are different colluvial phases indicated by variations in (residual) D_e?

3.1.3. Results

The polymineral fine fraction exhibited low sensitivity and scattered D_e values. However, there was an overall increase in D_e with depth, which contained similar fluctuations in IRSL, post IR OSL and post IR & OSL TL data through the stratigraphy. IRSL and post IR & OSL TL yielded similar D_e values; lower values were obtained using post IR OSL and are discussed below.

The polymineral coarse fraction exhibited higher sensitivity than the fines, and better reproducibility from the optical signals. IRSL and post IR OSL yielded similar D_e values. Higher values from TL may indicate unbleached residuals. There was little overall change in D_e with depth, but similar fluctuations in the IRSL and post IR OSL results.

The HF-etched coarse fraction exhibited the highest sensitivity and best reproducibility. D_e values were somewhat higher than the single 50 Gy regenerative dose used initially, so growth up to 150 Gy was measured and fitted, which yielded more scattered and slightly higher values due to the onset of saturation. Trends and fluctuations in D_e down section were similar to those observed from the polymineral coarse fraction using IRSL and post IR OSL.

OSL on the HF etched coarse fraction, and IRSL and post IR OSL on the polymineral coarse fraction yielded similar D_e values and exhibited similar, generally less than 10 Gy, fluctuations through the statigraphy. The inversion in D_e between layers 6 and 7 was reproduced in both the sections sampled, indicating that such small fluctuations were not artefacts and may be related to the presence of residual doses linked to phases of colluviation. However, when approximate "apparent age" estimates were calculated from these D_e values, based on field gamma dose rates assuming a β:γ ratio of 2:1, they were found to lie around 40 ka, which is consistent with independent age control.

3.1.4. Outcomes

The profiling results indicate that full dating of coarse quartz and/or feldspar separates from a small number of samples would be sufficient to tie in the profiling results and provide a detailed chronostratigraphy for this site. They also indicate that the lack of overall progression in D_e with depth will make differentiation between the ages of different layers dependant on dose rate determinations, and on the interpretation of fluctuations in residual level. Profiling indicates where residuals are lowest, and hence where populations of fully reset grains are more likely be identified in more detailed analyses.

3.2. Monasheskaya (Beliaeva, 1999)

3.2.1. Background

A limestone cave site in the Gubs Gorge, Northern Caucasus, Monasheskaya contains Middle Palaeolithic archaeology and pollen indicating fluctuating warm (wet) and cold (dry) spells in OIS stage 3 (Beliaeva, 1999). All levels are rocky cave deposits, some with evidence for burrows. Rockier horizons in Fig. 3(b) broadly equate with colder climatic stages. Layer 2 contained Neanderthal skeletal remains, layer 3a (3A-5) was a dark, ashy occupation layer (Fig. 3(b)).

3.2.2. Questions

Are there well-bleached layers or components in these deposits? Did frost action during cold periods or chemical weathering during warm periods introduce geological age grains from the limestone? Are externally derived sediments present? Has bioturbation, including human activity, affected the luminescence results?

3.2.3. Results

Across the mineral/grain-size fractions, D_e values were highest for IRSL, lower for post IR & OSL TL, and lower for OSL and post IR OSL, but residuals in the modern surface sample were close to zero for all the optical signals, and only ca. 10 Gy for the TL signals. The most consistently reproducible results were obtained from layer 3a (3A-5), layer 2, and the modern surface.

The polymineral fine fraction exhibited good reproducibility in the upper section, but poor in the lower part. The polymineral and HF-etched coarse fractions exhibited overall increases in D_e with depth, but the samples from layer 4 (Lower) and 3a (3A-5) yielded lower values than the samples nearest them. Taking into account variations in dose rate, the apparent age for layer 2 would also appear low compared with the sample from layer 3 (Upper).

The low apparent ages and better reproducibility observed for the artefact rich layers 2 and 3a (3A-5) indicate lower residual doses, and hence less contamination and/or more bleaching than rocky layers accumulating during colder periods. However, although the thin dark layer 3a (3A-5) did not appear grossly disrupted, the introduction of younger material by burrowing animals remains a possibility. Only post IR OSL on the polymineral fine fraction would yield apparent ages consistent with OIS 3.

3.2.4. Outcomes

Luminescence profiling has provided indications of residual levels and the significance of different sedimentary mechanisms at this site, to aid sample selection and provide context for the dating samples. Profiling indicates that the most promising samples for dating were from the most anthropogenic sediments, which are traditionally avoided in luminescence dating studies in favour of sterile (natural) layers with simpler formation mechanisms. The levels of scatter and residual dose observed in the sterile layers indicate that single grain analysis may be required for any of the samples, in order to improve significantly on the information already obtained by luminescence profiling.

3.3. Akhshtyr (Liubin, 1989)

3.3.1. Background

A limestone cave site in the Sochi region, North-west Caucasus. The Upper Palaeolithic levels at Akhshtyr are rocky cave sediments with γ-dose rates similar to those at Monasheskaya ($\sim$0.3 mGy/a). The Middle Palaeolithic levels are fine sediments with higher γ-dose rates ($\sim$0.8 mGy/a), and layer boundaries indicative of scouring by water (Fig. 3(c)). Layer 2 has been dated to 19 ± 0.5 BP by ^{14}C, and fallen stalagtites collected from layer 3a were U-Series dated to 35 ± 2 ka (Liubin, 1989).

3.3.2. Questions

Was the sediment in the lower levels washed from the back of the cave as suggested by Liubin (1989)? If so, how did it arrive there in the first place—might it have been washed in and potentially bleached, and if so when? Is there an externally derived component in the finer material of the upper levels, or is it dominated by (unzeroed) weathering products?

3.3.3. Results

The polymineral fine fraction exhibited very low sensitivity and hence highly scattered D_e values (Table S2; Fig. 3(c)). High D_e values were measured from the polymineral coarse fraction by regenerating the samples up to 500 Gy. IRSL yielded slightly lower values than post IR OSL and post IR & OSL TL. Trends for increasing D_e down-section simply mirrored changes in dose rate down section. Apparent ages for all samples were greater than 150 ka. The HF etched fraction exhibited increases in D_e with depth in the upper levels, but D_e levelled off around 140 Gy. Regenerating to higher doses indicated saturation around 150 Gy.

3.3.4. Outcomes

Luminescence profiling indicates that the sediments sampled from this site offer little potential to obtain archaeologically meaningful luminescence dates, as it supports the hypothesis of older material being reworked within the cave. The rocky cave sediments should therefore be the focus of any further investigations, but the lack of scatter or fluctuations to relatively low values of D_e, in the profiling data, indicates that the identification of age populations concordant with independent age control would be unlikely.

4. Discussion

When considering the interpretation of data generated following the luminescence profiling approach described above, one is presented with a number of technical issues.

Irradiation and measurement induce changes in luminescence sensitivity, particularly strongly in quartz (e.g. Stokes, 1994). This was accounted for in OSL measurements on the HF etched fraction by normalising to test dose response, but not in the multiple stimulation measurements on the polymineral fractions (Table S1). The IRSL signal, which is not expected to include a significant contribution from quartz, may sensitise differently and may also be more strongly diminished by any anomalous fading of the signal from feldspars (Wintle, 1977).

Changes in apparent sensitivity can also result from colouration: the aliquots of polymineral fine material prepared using the rapid protocol outlined above often darkened during the measurement procedure, which would probably cause a reduction in sensitivity and hence an overestimate of D_e. Doses absorbed by the polymineral fine fraction, and to a much lesser extent the (unetched) polymineral coarse fraction would be higher than the etched coarse fraction due to external alpha radiation and differences in internal radioactivity (Fleming, 1966; Zimmerman, 1971; Mejdahl, 1983; Huntley and Baril, 1997). Differences in the average α efficiency of the luminescent minerals in the fine-grain fraction could also contribute to differences in D_e between signals and samples.

For example, at Kostienki and Monasheskaya, IRSL on both *coarse* and *fine* polymineral fractions yielded similar or greater D_e values than post IR OSL, while post IR OSL D_e values for the polymineral *coarse* fraction were similar to sensitivity corrected OSL values for the etched coarse fraction. At both these sites OSL and post IR OSL sensitivity was high relative to IRSL (Table S2), indicating a strong quartz contribution to post IR OSL signals from the polymineral fractions. Therefore, quartz sensitisation and differences in anomalous fading of the IRSL and post IR OSL signals do not appear to have produced the observed similarities and differences. Roberts and Wintle (2001) observed similar differences between IRSL and post IR OSL results, in SAR measurements on polymineral loess samples. They investigated and discounted differences in α efficiency, relative bleaching, and thermal transfer effects, instead allowing for the possibility of desensitisation of the IRSL response during measurement of the natural signal. In the present study the desensitisation would need to vary from $\sim$0% to $\sim$90% between aliquots to account for the differences.

However, the point of luminescence profiling is that it is a basic survey approach that attempts to illustrate rather than to resolve such issues, for which more comprehensive measurement techniques have been developed. Bearing in mind the above, one may compare more or less directly between data within a given dataset, e.g. in terms of residuals. Qualitative comparisons may also be made between datasets from different stimulation methods and/or mineral/grain-size fractions, e.g. in terms of patterns and reproducibility. It is notable though, that despite the issues outlined above there is often broad quantitative agreement between profiling results obtained using different stimulation methods, and on different mineral/grain-size fractions from a series of samples (e.g. Fig. 3(a)).

5. Conclusions

This study represents the latest step in optimisation and application of the luminescence profiling approach to site investigation. It demonstrates how the profiling approach can contribute to efficient assessment of a site and/or sediment's suitability for luminescence dating, and help to select samples and methods for more intensive analysis. Further, luminescence profiling has enhanced understanding of sedimentary process at these sites, using its spatial resolution to identify changing environmental conditions in a way not possible with intensive analysis of smaller numbers of samples.

Acknowledgements

The authors would like to thank Prof. V.P. Liubin, Dr. E.V. Beliaeva, and Dr. A.A. Sinitsyn, for their great help and advice in relation to the field sites and sampling. The assistance of Mr. D. Bogdanov in the field is gratefully appreciated. Thanks to Dr. A. Cresswell for preparing Fig. 1. This project is funded by the UK Natural Environment Research Council (NERC), through the thematic programme, "Environmental Factors in the Chronology of Human Evolution and Dispersal" (EFCHED).

Editorial handling by: R. Roberts

Appendix A. Supplementary data

The online version of this article contains additional supplementary data. Please visit doi:10.1016/j.quageo.2006.05.024.

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