Optically stimulated luminescence dating of Palaeolithic cave sites and their environmental context in the western Mediterranean

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6. Conclusion

The purpose of this thesis was to establish reliable chronostratigraphies for Palaeolithic cave sites in the western Mediterranean. The data produced by this work was then combined with archaeological, geological and sedimentological evidence from the sites to gain insights into hominin subsistence strategies, as well as hominin interaction with changing palaeoenvironments throughout the Pleistocene. Consistent application of single-grain quartz OSL to sites of varying antiquity addresses two main themes. The first is investigation of the general suitability of these quartz grains as luminescence chronometers and how variable their luminescence properties are on site and regional scale. The second informs us how the timing and geographical distribution of changes in Palaeolithic human behaviour and occupation patterns in the region might be linked to the different population dispersal events within Africa and into Europe that took place over the last ~2 Ma. OSL dating of individual sand-sized grains provides valuable information on the sedimentary history of sampled deposits that contribute to an improved understanding of local site formation processes and past palaeoclimatic variability in the area.

6.1. Quartz luminescence dating

Quartz OSL dating has increasingly been used to determine the burial age of sediments at Palaeolithic cave sites in the western Mediterranean over the past two decades. Most of those studies applying multiple-grain dating approaches (e.g. Álvarez-Alonso et al., 2016; Barton et al., 2009; Galván et al., 2014; Ramos et al., 2008; Rhodes et al., 2006; Schwenninger et al., 2010). Single-grain dating of sand-sized quartz grains at archaeological sites in the region, on the other hand, was primarily adopted to small subsets of samples to supplement the results of other radiometric dating techniques - e.g. at Taforalt (Clark-Balzan et al., 2012), Morocco, or Cuevá Anton (Zilhão et al., 2016) and Gorham's Cave (Rhodes, 2013), both in southern Iberia. Complete chronostratigraphies have so far only been generated for three coastal sites in the Témara region, Morocco (Jacobs et al., 2011; Jacobs et al., 2012). Consequently, our knowledge on how individual grains of quartz from these areas behave during luminescence dating is limited. Single-grain data elucidate the potential of multiple-grain measurements to under- or overestimate the true age. The luminescence characteristics of the deposited sediment itself or locally occurring site-specific processes, like i.e. post depositional intrusion of grains or carbonate cementation – had yet to be studied systematically at single-grain level.

Within this thesis, the investigated individual quartz grains from three western Mediterranean archaeological cave sites were shown to exhibit generally bright, fast-component-dominated luminescence signals. Initial standard OSL performance tests proved the material generally suitable for measurements using the SAR protocol and single grain rejection criteria were successfully applied to discard aberrant grains from the D_e datasets.

Despite showing promising prerequisites for OSL dating, there are also some considerable differences recognisable in the luminescence characteristics of the sediments between sites. Among these are the low proportions of individual grains from Vanguard Cave, Gibraltar (chapter 5), exhibiting luminescence signals suitable for D_e determination (7–15%) compared to the Moroccan sediments (12-62%). The majority of grains from Vanguard Cave giving measurable luminescence signals also pass other rejection criteria, which contrasts with the grains investigated at Rhafas (chapter 2) and Thomas Quarries (chapter 3). The latter regularly fail quality criteria due to signal saturation, poor recycling ratios, or feldspar contamination. Luminescence characteristics of the sediments in this thesis, furthermore, vary through time at a given site. Although a few extremely bright grains with above-average D_e values occur in low proportions in some samples from Vanguard Cave and might have an impact on determined ages when using multiple-grain aliquots, the sediments are overall characterised by homogeneous luminescence behaviours. This is the opposite of what was observed at Rhafas, where causes for individual grain rejection are highly layer-dependent. Quartz grains from the Thomas Quarries are not only affected by luminescence signal saturation. Single-grain dose recovery tests from that site yielded substantial dose recovery ratio underestimations and high intrinsic overdispersion (σ_{OD}) values. These results question the ability of the material to recover known laboratory doses, as well as the applicability of dose recovery tests which are commonly based on multiple-grain measurements.

In chapter 4 of this thesis, we examine whether dose recovery tests are a useful check for i) the suitability of the measured SAR protocol and ii) the reliability of natural D_e estimates in single-grain dating of archaeological sediments. Recently, some authors have cast doubt on the significance of such tests. They found no correlation between dose recovery ratios and accuracy of single-grain age estimates (Guérin et al., 2015) and showed that the size of the given dose in such experiments has considerable impact on the test results (Thomsen et al., 2012). Dose recovery characteristics of samples from Thomas Quarry I and Rhafas (natural D_es >180 Gy) following different bleaching methods (sunlight, green laser beams and solar simulator) and sizes of administered doses were systematically investigated. The results were compared to those derived from highly sensitised, well-bleached Australian quartz sand with comparatively small natural D_es from the site of Lake Mungo (Fitzsimmons et al., 2014) using the same test parameters. While the Australian samples show primary dependencies of dose recovery ratios and σ_{OD} on the size of the given dose, the Moroccan samples yield more variable results. No obvious correlations exist between the bleaching treatments or the size of the given dose, and the accuracy and precision of recovered doses in two out of three Moroccan samples. Thus we concluded that – in addition to administered dose size - sample-specific responses to chosen test parameters can significantly influence single-grain dose recovery test ratios and σ_{OD} values, particularly in less sensitised archaeological cave sediments like Thomas Quarry I and Rhafas. Performing a dose recovery test to assess the validity of the chosen SAR protocol parameters in these samples can lead to highly controversial results depending on the size of the given dose and on the bleaching source used. It remains doubtful as to which extent such tests can serve as quality check of the measurement protocol and the reliability of final age estimates.

The overdispersion value, which quantifies the spread in D_e distributions, is commonly used as an argument to justify the application of specific statistical age models in single-grain dating studies. Olley et al. (2004) stated that single-grain D_es of well-bleached natural samples are typically overdispersed by 9-22%, while σ_{OD} values vary between 7-12% during laboratory-based dose recovery tests, whereby all extrinsic factors influencing the spread of natural D_e distributions are excluded (Jacobs et al., 2006; Reimann et al., 2012; Thomsen et al., 2007). Intrinsic σ_{OD} in dose recovery experiments in this thesis (chapter 4) is highly variable for both Australian and Moroccan samples – 5-27% and 6-35%, respectively. These values may even exceed the value determined for the natural D_e of a sample. Thus, definite

statements as to the amount of intrinsic σ_{OD} in natural D_e distributions and a correct assessment of the extrinsic component – which is usually associated to dose rate heterogeneity, incomplete bleaching or post depositional mixing – cannot be reliably made for our samples. Examples like these and studies like Guérin et al. (2015) or Thomsen et al. (2012) raise questions concerning the implication of σ_{OD} values, the impact of natural and/or experimental factors influencing them and whether they can serve as arguments justifying the use of statistical age models in single-grain dating. Consequently, decisions regarding the application of CAM or FMM for the samples from Vanguard Cave, Rhafas and the Thomas Quarries were primarily made based on the shape of single-grain D_e distributions, as well as sedimentological and stratigraphical observations in field, rather than σ_{OD} values.

Challenges for single-grain dating at the investigated cave sites are predominantly a result of sediment composition and local site formation processes that lead to heterogeneous dose rates and post-depositional mixing in the stratigraphical layers. Partial bleaching of the luminescence signal stored in the sand grains was not observed at any of the three sites. While at Rhafas (chapter 2), a single mixing event significantly affects D_e distributions of three layers in the cave mouth section. One main source of uncertainty for reliable age determination at all sites relates to correct assessments of the environmental dose rates of each layer. At Vanguard Cave (chapter 5), patches and small layers of potassium-rich clays, which are known to increase dose rates, commonly occur throughout the stratigraphical profile. Carbonates, on the other hand, which decrease the dose rate of a sediment layer, are present at all sites in form of exogenic carbonate fragments from local limestone bedrock, crusts/layers/lenses of pure carbonate, and post-depositional cementation of sediment layers. Consequently, stratigraphical profiles at these cave sites are characterised by inhomogeneous sediment layers with highly variable beta and gamma dose rates. Variations in microdosimetry are, furthermore, likely to influence equivalent dose at single grain level. Moreover, introduction of allochthonous radioactive elements via groundwater after sediment deposition was identified for the lower cave section at Rhafas and renders these layers undateable using standard quartz OSL dating. An upcoming project will clarify whether subtraction dating (Feathers, 2002) or isochron dating (Li et al., 2008) can overcome those dose rate issues and determine the age of the lower cave section sediments at Rhafas.

In conclusion, for western Mediterranean cave sites, it is highly recommended to analyse site formation and sediment composition in great detail and to use multiple methods for dose rate determination – including in-situ measurements – to identify potential mixing processes and to be able to assess the dose rate of each dated layer.

6.2. Implications for Palaeolithic human behaviour and dispersal

In this thesis, single-grain OSL dating was successfully used to establish reliable chronological frameworks for stratigraphical profiles at two Palaeolithic cave sites in the western Mediterranean: Rhafas (chapter 2) and Vanguard Cave (chapter 5).

For the sites of Thomas Quarry I and Rhino Cave near Casablanca (chapter 3), however, standard quartz OSL dating of individual grains did not yield conclusive results for age determination. Large numbers of single-grains at Casablanca are affected by luminescence signal saturation; others pass all established quality criteria, while still failing to correctly recover known laboratory doses. D_e distributions are widely scattered and calculated absolute ages lack stratigraphical consistency, irrespective of the applied statistical age model. While the latter could at least partially result from the fact that present-day dose rates do not correctly reflect average burial-time dose rates within the sampled layers - since large parts of the stratigraphical profiles experienced intensive post-depositional cementation by

carbonates – the former reinforces doubts as to the general suitability of these sediments for standard quartz single-grain dating. Comparison with independent age control available for the Thomas Quarries shows that calculated single-grain ages suffer from substantial age underestimation. Given the successful OSL dating results from Rhodes et al. (2006) and the dose recovery experiments performed in this thesis (chapter 4), it can be stated that luminescence dating is not necessarily an inadequate technique for age determination of the Thomas Quarries sediments. While individual grain dating using a standard SAR protocol did not result in reliable ages for both sites, sediments are nevertheless extremely promising for future research focussing on other luminescence approaches such as thermally transferred or violet stimulated OSL. The single-grain dating study performed at the Thomas Quarries in this thesis, unfortunately, does not allow any conclusions regarding the role of northern African populations in the emergence of the European Acheulian or in the debate of potential ancient human crossings of the Strait of Gibraltar during the Early Pleistocene. It nevertheless highlights once more the fundamental importance of cross-checks between different Quaternary dating techniques for reliable age determination of Palaeolithic caves sites.

The long stratified sequence at Rhafas (chapter 2) covers a time period from >135 ka to the Neolithic (~7.8 ka), during which two important human dispersal events from Africa into Europe took place: Out of Africa 2a and 2b, in MIS 5 and MIS 3, respectively. The archaeological record of the site comprises classical MSA, Aterian, LSA as well as Neolithic industries spread over three sections within the cave itself and on a flat terrace area in front of the cave opening. Evidence for human occupation of the Maghreb during the Middle Pleistocene predating MIS 5 is extremely sparse and limited to a few sites in Morocco, Tunisia and western Libya. Recent studies at Jebel Irhoud, western Morocco, emphasise the significance of this region for understanding the emergence of the human lineage in Africa by presenting reliable evidence for early *homo sapiens* fossil remains (Hublin et al., 2017) associated with MSA lithic artefacts as early as 315 ± 34 ka at the site (Richter et al., 2017). The archaeological record from Jebel Irhoud, therefore, predates the earliest appearance of MSA assemblages in the Maghreb – formerly associated with the Benzù rockshelter at 254 ± 17 ka in northern Morocco (Ramos et al., 2008; Ramos Muñoz et al., 2007) - by ~60.000 years. Its fossil remains, together with the partial cranium from Florisbad (Grün et al., 1996), South Africa, now represent the earliest known representatives of the *homo sapiens* clade. Archaeological finds proving human presence in the Maghreb become more frequent with the onset of MIS 6, when climatic conditions in Northern Africa were relatively dry. At Rhafas this time period is reflected by evaporite enriched sediments in the lower cave section containing MSA stone tools predating 135 ka. With the onset of MIS 5, humidity generally increased in Northern Africa, facilitating AMH expansions in the region, especially during the 'green Sahara' events in MIS 5a, 5c and 5e. Rapid peopling of all parts of the Maghreb can be easily traced through the substantial increase of archaeological sites ~130 ka, which is around the same time when hominin groups started to settle out of the African continent again (Out of Africa 2a). While a long-term colonisation of the Levant by AMHs failed, the Maghreb became permanently inhabited. MIS 5 deposits at Rhafas firstly reflect a change in sediment source compared to the underlying MIS 6 layers, which is likely to be linked to a regional shift in prevailing wind directions and, secondly, witness the transition from classical MSA to Aterian through the regular appearance of pedunculated tools after 123 ka. This lithic technocomplex is typically interpreted as indicator for behavioural modernity – a potential key factor to understand the evolutionary success of our lineage over Neanderthals - in AMH populations of the Maghreb. Its earliest reliable occurrence dates to 145 ± 9 ka at Ifri N'Ammar (Richter et al., 2012), northern Morocco.

A regional comparison of the data available today from dated archaeological sites in the Maghreb shows i) that tanged pieces became a widespread feature in archaeological assemblages with the onset of MIS 5e in Morocco, while being present in Algeria, Tunisia and western Libya not before ~65 ka,

and ii) that MSA and Aterian lithic assemblages coexisted in the region over a long time until they got eventually replaced by LSA industries in MIS 3. Future research must clarify whether the technological innovations of the Aterian truly emerged in Morocco or whether this temporal signal in the regional comparison is simply an artefact of the limited chronological data available for archaeological sites in other parts of the Maghreb. Likewise still unresolved questions which require further investigations in the region concern i) the debate as to whether the Aterian represents a phase within the MSA or has to be seen as separate entity in North Africa (Dibble et al., 2013; Iovita, 2011) and ii) the clarification of the role of Aterian people in the successful Out of Africa event 2b that took place after 60 ka.

The site of Rhafas (chapter 2), additionally, yields evidence for an intensive phase of human occupation between 21 ± 2 ka and 15 ± 1 ka associated with LSA lithic industries and for the prevailing palaeoclimate during MIS 3 and MIS 2. Climatic conditions at that time favoured intensive carbonate formation at the site including beside cementation of sediment layers in the cave mouth section a prominent duricrust in the terrace section. Detailed investigations of the duricrust fabric suggests that its formation took place over a longer period of time and under various climatic conditions ranging from relatively humid to arid but not hyper arid, just as Northern Africa experienced over MIS 3 and MIS 2. While the LSA appears in lithic assemblages – especially in South Africa – before 42 kcal BP at some sites (Ambrose, 1998; Villa et al., 2012), it occurs relatively late in the Maghreb. The regional comparison shows that early LSA assemblages are particularly widespread in western Libya between 35-30 ka, while they are not present – with the exception of Dar es-Soltan II, Morocco (Schwenninger et al., 2010) – in other parts of the Maghreb before ~24 ka. While the origins of the LSA in Africa are still matter of ongoing scientific debate, there also remains the question whether this technocomplex was brought to the Maghreb by AMH populations traveling through Northern Africa from the East to the West.

In chapter 5 of this thesis, a high-resolution dual chronostratigraphy for the uppermost ~4 m of the >17 m sedimentary sequence at Vanguard Cave, Gibraltar, was established using single-grain and multiple-grain OSL dating. Located at the present-day shoreline of the Mediterranean Sea and accumulated on top of a MIS 5 fossilized marine terrace, the site contains evidence for repeated occupation by Neanderthals at a time before AMHs arrived in Southern Iberia. Due to the fact that relatively few individual grains at Vanguard Cave emit sufficient luminescence signals for dating, averaging effects in 1 mm multiple-grain aliquots are almost negligible, resulting in an excellent correspondence between their dating results with those obtained using single-grains. Dated to between 66 ± 6 ka and 43 ± 3 ka, the uppermost ~4 m of the sedimentary profile are a reliable proof of the rapid aeolian accumulation that took place during MIS 3 and MIS 4 at the site, and which is likely to have also persisted throughout MIS 5.

Recent studies from Gibraltar indicate mild and sub-humid Mediterranean climatic conditions for the time of Neanderthal occupation, MIS 5-3 (Blain et al., 2013; Stringer et al., 2008), and the presence of an extensive – now submerged – coastal shelf on the east side of the Rock, directly in front of the Vanguard Cave entrance, covered with savannah and aeolian sand dunes during MIS 3 (Rodríguez-Vidal et al., 2013). Geomorphic evidence from the site presented in this thesis, indicates similar palaeoenvironmental conditions in the region with strong easterly winds throughout the entire depositional history of the sedimentary sequence. Ongoing and future research on the Vanguard Cave record coupled with the reliable chronostratigraphic framework offers great potential to significantly improve our understanding of climatic and environmental conditions as well as Neanderthal lifestyle in southern Iberia during MIS 5-3. Detailed analyses of the stone tools recovered from the uppermost layers of the cave fill sequence – previously described as Upper Palaeolithic (Barton et al., 2013) - must clarify their cultural affiliation within the Palaeolithic.

In conclusion, this thesis underlines that dating of archaeological cave sites using OSL remains a

highly complex task. In the western Mediterranean region, where luminescence characteristics of sediments are diverse and variable climatic conditions throughout the Pleistocene can largely affect sedimentological profiles post-depositionally, the use of individual grain dating is highly recommended, although even this method comes with caveats. Palaeoenvironmental signals can be stored at cave sites in the region enabling conclusive statements regarding climatic changes on local or regional scales. Reliable chronologies for Palaeolithic cave sites are crucial to understand human behaviour and dispersal patterns. They are, however, only one part of a complex picture and need to be complemented by archaeological, geological and sedimentological investigations to achieve their highest possible information value.

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