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Published

2022

Journal Title

Marine Geology

Version

Accepted Manuscript (AM)

DOI

10.1016/j.margeo.2021.106692

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Investigating the resetting of IRSL signals in beach cobbles and their potential for rock surface dating of marine terraces in Northern Chile

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Abstract

Inactive shorelines represent valuable records for sea level change, shoreline variations and tectonics if we can constrain the timing of their formation. Where the associated beaches are cobble dominated, luminescence rock surface dating is a promising alternative to established dating approaches, since unlike other techniques it offers the potential to identify clasts unaffected by inherited ages. While luminescence rock surface dating has successfully been used on Holocene and Late Pleistocene beach ridges previously, in this study the potential of IRSL rock surface dating is evaluated for the magmatic cobbles of uplifted Pleistocene terraces along the tectonically active coast of northern Chile. Cobbles from an active beach were used to investigate the influence of cobble lithology on IRSL signal properties and the effectiveness of IRSL signal resetting in the rock. While alkaline and andesitic cobbles yield low IRSL intensities and limited signal resetting due to strong light attenuation, more favourable characteristics for dating were observed for some diorite and granite cobbles. Their IRSL signals were well reset in the uppermost few mm without any systematic difference between upper and lower surface. Some of them revealed bleaching plateaus with inherited ages close to zero after correction for laboratory residuals. For dating, cobbles from three Pleistocene marine terraces, for which new uranium-thorium and ESR control ages on molluscs provide age control, were targeted. None of the associated IRSL rock surface burial ages agrees with the MIS 5 control ages of the terraces. Most of the selected cobbles are either too dark to allow for effective signal resetting or yield IRSL properties unsuitable for dating. Only one of the targeted cobbles shows both signs of signal resetting at its surface and sensitive IRSL signals, but its signal was already in field saturation due to dose rates >6 Gy/ka. In conclusion, our data indicate that beach cobbles with granitic to dioritic lithology combine appropriate IRSL properties and sufficient IRSL signal resetting for dating Holocene landforms. Last interglacial terraces may already be beyond the limit of IRSL dating for most cobbles of this lithology since they show large dose rates compared to IRSL sediment dating.

Keywords: Luminescence dating, rock surface dating, post-infrared-infrared, lithology, marine terrace, beach cobble, modern analogue, Atacama.

1. Introduction

While luminescence dating is typically used to determine the depositional age of sand- or silt-sized sediments, recent developments have extended its applicability to the dating of rock surfaces. Based on the observation of time- and depth-dependent luminescence signal resetting in the uppermost millimetres to centimetres of light-exposed rock surfaces (Habermann et al., 2000; Sohbati et al., 2011), luminescence rock surface dating has shown potential for quantifying both burial and exposure durations of rock surfaces (see King et al., 2019b for a review). This approach offers the potential to apply luminescence dating to new substrates and research questions, including the formation of moraines (Jenkins et al., 2018; Rades et al., 2018), rock falls (Sohbati et al., 2012), archaeological structures and artefacts (Freiesleben et al., 2015; Sohbati et al., 2015; Ageby et al., 2021; Gliganic et al., 2021) and coastal boulders (Brill et al., 2021), as well as the reconstruction of deglaciation histories (Lehmann et al., 2018, 2020) and erosion rates (Sohbati et al., 2018; Lehmann et al., 2019). Luminescence rock surface dating may also inform about the depositional history of wave-transported beach cobbles (Sohbati et al., 2011). When forming raised beaches or marine terraces, beach cobbles can mark the position of former shorelines and, thus, may serve as indicators for sea-level variations (e.g. Garrett et al., 2020), rates of tectonic uplift or subsidence (e.g. Merrits and Bull, 1989), and shoreline displacements (e.g. Brill et al., 2015). Chronologies based on radiocarbon, electron spin resonance (ESR), U-series or amino acid racemization dating of calcareous organisms incorporated in beach deposits are relatively common (e.g. Ortlieb et al., 1996; Schellmann and Radtke, 2000), but may be biased if the dated organisms have been reworked from older deposits, or behaved as open systems for U-series elements, and especially if they have experienced uranium leaching. In absence of sandy sediment suitable for conventional luminescence dating, terrestrial cosmogenic nuclide dating is the most common approach for determining the formation of marine terraces like wave-cut platforms or cobble deposits (e.g. Saillard et al., 2009). As first successful applications have indicated (Simms et al., 2011, 2012; Simkins et al., 2013; Souza et al., 2019, 2021), luminescence rock surface dating can provide complimentary chronological constraints for cobble beaches over at least Holocene and Late Pleistocene time scales; for specific lithologies such as pure quartz clasts, the approach may even date back to MIS 12 (Bailiff et al., 2021). Luminescence rock surface dating may also be advantageous compared to conventional luminescence dating where sandy strata are available since depth-resolved luminescence data offer the opportunity to unambiguously identify completely bleached deposits and dose rates virtually unaffected by temporal changes of water contents (Jenkins et al., 2018).

The aim of this study is to systematically evaluate luminescence rock surface dating applied to raised beaches and Pleistocene marine terraces along the tectonically active coast of Chile, which serve as valuable indicators for regional tectonics and relative sea-level fluctuations. Since quartz luminescence properties of magmatic rocks, as they dominate the lithology of the Andean Cordillera, are typically inadequate for dating (e.g. Tsukamoto et al., 2011; Simkins et al., 2016), we applied feldspar infrared stimulated luminescence (IRSL) and post-infrared-infrared stimulated luminescence (post-IR-IRSL) rock surface dating to cobbles from a modern beach and three Pleistocene marine terrace levels along the coast of northern Chile. Firstly, we studied the influence of different cobble lithologies on the associated IRSL and post-IR-IRSL signal properties. Secondly, the effectiveness of IRSL and post-IR-IRSL signal resetting at cobble beaches was investigated by using cobbles from a modern beach. And thirdly, cobbles from two Pleistocene marine terraces, for which chronological control is available from conventional post-IR-IRSL sediment ages (Bartz et al., 2020a), were targeted with IRSL and post-IR-IRSL rock surface burial dating. As additional age cross-check, we provide new U-series and combined U-series/ESR ages of three mollusc shells from the same sediment sections. The benefits of this study are not restricted to marine terraces along the coast of northern Chile, they are assumed to provide valuable insights into the potential of luminescence rock surface dating techniques to date marine terraces and raised cobble beaches in general.

2. Study area

Our study area is located at the coastal Atacama Desert (N Chile) between Iquique and the Mejillones Peninsula (Fig. 1a). The main geomorphological structures are the Coastal Cordillera and the Coastal Plain. The former is an eroded volcanic arc formed in the Mesozoic, mostly ~1000-2000 m high and on average ~30 km wide (González et al., 2003). Its western margin is characterised by the Coastal Cliff. Regard et al. (2010) estimated the elevation and timing of the cliff foot to ~110 m a.s.l (above sea level) and ~400 ka, respectively. The up to 3-km wide Coastal Plain between the modern coastline and the Coastal Cliff exhibits hundreds of alluvial fan sequences (Walk et al., 2020) overlying and intersecting with uplifted marine terrace deposits (e.g. Radtke, 1989; Bartz et al., 2020b). In the tectonically active setting of the Andean subduction zone, marine terraces provide excellent indicators for tectonic uplift and the regional relative sea-level history (e.g. Ortlieb et al., 1996; Regard et al., 2010). A significant number of studies have conducted numerical dating to chronologically constrain the marine terrace deposits, including luminescence and ESR dating of sediments, ESR in combination with U-series dating of marine shells, as well as amino acid racemization and radiocarbon dating of shells and terrace material (Bartz et al., 2020a, 2020b; González-Alfaro et al., 2018; Labonne and Hillaire-Marcel, 2000; Leonard and Wehmiller, 1991; Ortlieb et al., 1996; Radtke, 1989; Ratusny and Radtke, 1988). These studies resulted in an almost complete chronology of palaeo-shorelines from the Holocene to MIS 11 (Regard et al., 2010). Quaternary uplift rates in the area are between 0.1 and 0.6 m/ka (Victor et al., 2011), but have likely varied spatially and temporally during the Quaternary (e.g., Binnie et al., 2016; Saillard et al., 2009). This is indicated, for example, by varying elevation levels between 10 and 60 m a.s.l. for MIS 5 marine terrace surfaces (Radtke, 1989).

The sediments forming alluvial fans and marine terraces are mainly derived from fluvial systems that cut back into the Coastal Cordillera (Walk et al., 2020). The catchments feeding the fan-terrace complexes studied here are mainly characterized by (i) Mesozoic plutonic rocks with granitic to dioritic composition (specifically the Cerro Carrasco complex, the Gatico complex and the Tocopilla complex), and (ii) volcanic rocks of the Jurassic La Negra formation mainly composed of andesites and basalts, and only to a minor part of (iii) unconsolidated Late Tertiary sediments (e.g. the Alto Hospicio gravels) (Medina et al., 2012; Quezada et al., 2012; Sepulveda et al., 2012; Vasquez and Sepulveda, 2012; Mpodozis et al., 2015). Due to the long-term hyper-aridity of the region with currently less than 5 mm of annual rainfall on average, which was intersected only by short periods with slightly more humid conditions during the Pleistocene (e.g. Ritter et al., 2019), preservation conditions for raised coastal deposits and landforms in the study area are generally good.

3. Material and methods

3.1 Site stratigraphy and sample selection

Beach cobbles for luminescence rock surface dating were collected at three locations along the Atacama coastline, namely Río Seco (SEC), Guanillos (GUA) and Caleta El Fierro (VIR – “Virgen del Camino”) (Fig. 1a, Tab. S1). The geomorphological settings of all three sites have previously been described in Bartz et al. (2020a, 2020b) and Walk et al. (2019, 2020).

Río Seco (SEC)

The Río Seco site is characterised by an alluvial fan complex that intercalates with at least one level of marine terrace deposits (Figs 1b,c; Bartz et al., 2020a,b). The marine sediments are composed of a succession of sandy units, layers with well-rounded cobbles and shell layers (Fig. 2a). At an elevation of ~21 m a.s.l. (section S4), a palaeo-beach composed of cobbles in sandy matrix was sampled for luminescence rock surface dating (SEC 4-3). Sandy beach deposits directly above this cobble layer were dated to older than 110 ka based on post-IR-IRSL$_{225}$ dating of potassium feldspar (Bartz et al., 2020a). For additional cross-check, two well-preserved, closed bivalves (SEC 4-2 and SEC 4-2a) from an up to 2 m thick mollusc layer directly above the sandy beach facies were sampled for ESR and U-series dating (Fig. 2a). Due to its heterogeneous composition of well-preserved bivalves of different species, shell fragments, rounded beach cobbles and angular alluvial fan clasts, as well as its lateral continuity over more than 200 m, the mollusc layer might be related to flooding by a high-energy event such as a tsunami rather than beach processes (layers with similar characteristics were interpreted as Holocene tsunami deposits in the South of the Atacama by León et al., 2019). Approximately 150 m seaward of section S4, beach deposits at an elevation of ~12 m a.s.l. (section S2 in Fig. 1c) were dated to the MIS 5c (95-110 ka) based on a combination of potassium feldspar post-IR-IRSL and quartz ESR dating (Bartz et al., 2020a). Based on the existing geochronological framework (Bartz et al., 2020a), as well as on observations by Radtke (1989) from marine terrace deposits close to Iquique, the marine deposits of S2 and S4 likely correspond to different MIS 5 sea levels (S2 was formed during MIS 5c and S4 during MIS 5e or older).

Guanillos (GUA)

The Guanillos alluvial fan complex is fed by two fluvial catchments. Marine terrace deposits have not been observed at this location, most likely due to superposition or erosion by the Late Pleistocene alluvial fan activity (Bartz et al., 2020b). Cobbles for luminescence rock surface dating were collected from the modern beach profile south of the main alluvial fan outlet (Figs 1d,e). All cobbles originate from the seaward side of the present beach berm. Surface samples were collected in elevations of ~1 m a.s.l. (GUA 3), ~1.5 m a.s.l. (GUA 2) and ~2 m a.s.l. (GUA 1).

Virgen del Camino (VIR)

At the alluvial fan complex at Virgen del Camino (VIR, also called Caleta El Fierro), a succession of palaeo-beach deposits composed of well-rounded cobbles and homogeneous fine- to medium-sand with abundant shell fragments and few mollusc shells is exposed along the outlet of the main channel (Bartz et al., 2020a,b; Figs 1f,g, 2b). Cobbles for luminescence rock surface dating were extracted from a beach-cobble layer at ~17 m a.s.l. (VIR 3-1) that was exposed at the northern channel section (V2a in Fig. 2b). Bartz et al. (2020a) post-IR-IRSL dated sandy upper shoreface deposits below (~16 m a.s.l.) and sandy beach deposits above the cobble layer (18-20 m a.s.l.) to the MIS 5e (120±10 ka) and MIS 5a/c (~90 ka), respectively (Fig. 2b). For their interpretation it is important to highlight that the MIS 5e deposits were deposited under shallow marine conditions, so the contemporaneous palaeo-beach deposits might be buried further inland at higher elevations (Leonard and Wehmiller, 1991; Bartz et al., 2020b). As a further crosscheck, a shell (VIR 2-1) from the sandy beach deposits above the cobble layer was collected for ESR and U-series dating at the southern channel section (V2c in Fig. 2b), where its location is laterally concordant to the post-IR-IRSL$_{225}$ dated sand horizon (Bartz et al., 2020a). A second cobble sample for luminescence rock surface dating (VIR 3-2) was collected from the landward cliff of a wave-cut platform at ~7 m a.s.l. (section V3, Figs 1f,g, 2b). The ~10 m high cliff at the alluvial fan toe and the associated wave-cut platform were formed during the Holocene, as indicated by ^{10}Be cosmogenic nuclide ages of ~10 ka (Bartz et al., 2020b; Fig. 2b). The cobbles collected for dating originate from a wave-cut notch at the cliff foot. Deposition of these cobbles could be both synchronous to the formation of the Holocene wave-cut platform, or to the formation of the MIS 5 marine strata exposed at the 8-m high knick-point of the alluvial channel outlet.

3.2 IRSL rock surface burial dating

Cobbles from the modern beach (GUA) were collected directly at the surface of the beach profile, while those from the marine terraces (VIR, SEC) were sampled at natural outcrops after removing the outermost ~30 cm of cobbles (Fig. 3a). After marking shielded and exposed cobble surfaces at the time of sample collection, cobbles were sealed in light-proof plastic bags for laboratory analyses.

Further processing of the cobbles was performed under subdued red light conditions in the Cologne Luminescence Laboratory (CLL) to avoid any bleaching of luminescence signals after sample collection. For each of the six samples, two cobbles were prepared (Tab. S1). Rock cores of ~1 cm diameter and 2-4 cm length were extracted from the cobbles using a water-cooled bench drill (WEKA DK17) with diamond core bits. In case of the modern beach samples (GUA), one core was extracted from the surface exposed during sampling and a second one from the shielded bottom surface. Only cores from the surface shielded during sample collection were taken from the marine terrace cobbles at VIR and SEC (Fig. 3b). All cores were cut into ~0.7 mm thick slices (in intervals of 1 mm) using a water-cooled low speed saw (Bühler Isomet 1000) with 0.3 mm thick diamond blades (Fig. 3c).

Luminescence measurements with a Risø TL/OSL DA 20 reader (Bøtter-Jensen et al., 2010) were performed on the complete rock slices or, for broken slices, on fragments put in aluminium cups without any previous chemical treatment or mineral separation. Since the measurement of polymineralic rock slices and fragments with significant feldspar contents does not permit robust instrumental isolation of quartz luminescence signals (Aitken, 1998), and since previous studies indicated inadequate luminescence properties for quartz from magmatic rocks in general (Sohbati et al., 2011; Souza et al., 2019) and quartz from Chile in particular (del Rio et al., 2019; Brill and Cisternas, 2020), we focused on feldspar luminescence signals in all our measurements. All measurements followed the post-IR-IRSL$_{225}$ protocol (cf. Buylaert et al., 2009) outlined in Table S2 using infrared LEDs for signal stimulation and a photomultiplier plus a LOT interference filter with peak transmission at 410 nm for signal detection. All signals are based on integrating the first 10 seconds of each signal curve and subtracting the last 20 seconds. It is one of the standard protocols for Pleistocene sediments in general, which was already used successfully in numerous conventional luminescence dating studies on silty to sandy sediments from the study area (e.g. Bartz et al., 2020a,b; May et al., 2020; Medialdea et al., 2020).

The measurement strategy outlined in Figure 3c started with the determination of luminescence signal-depth profiles by measuring L_n/T_n ratios for the uppermost 10-20 slices of each core. Dose recovery experiments with given doses of ~30 Gy and the quantification of laboratory residual doses were performed on 24 hours solar simulator (Hoenle Sol2) bleached slices from the inner part of each cobble using the full post-IR-IRSL$_{225}$ protocol. Rock slices for equivalent dose (D_e) determination were selected from those parts of the cobbles that were indicated as bleached by the associated signal-depth profiles (typically from the uppermost 1-3 slices, see sections 4.3 and 4.4) and measured using the full post-IR-IRSL$_{225}$ protocol (Tab. S2). Successive data analysis was performed separately for both luminescence signals measured within our protocol, the IRSL signal and the post-IR-IRSL$_{225}$ signal.

For dosimetry, the combined dose rates of the feldspar crystals (internal dose rate), the cobbles and the surrounding sediments (external dose rates), as well as cosmic radiation (cosmic dose rate) were considered (Figs 3b, c). Internal dose rates were calculated on the basis of assumed potassium concentrations of 10±2 % in potassium feldspar (Smedley et al., 2012) and 3±2 % in plagioclase (Sohbati et al., 2013), as well as average crystal sizes deduced from visual inspection of rock slices under a microscope. Cosmic dose rates were based on the geographic location and burial depth (Prescott and Hutton, 1994). Infinite matrix dose rates of the dated cobbles and the surrounding fine sediment were based on uranium, thorium and potassium contents determined with high-resolution gamma spectrometry. For the cobble matrix surrounding all dated cobbles, we assumed homogeneous lithology calculated by averaging the dose rates of all dated cobbles from a section, as well as pore volumes of 30 % that were filled with sand at VIR 3-1 and SEC (the associated sand dose rates were reported by Bartz et al., 2020a) and filled with air at GUA and VIR 3-2. We did not consider differences between the buried bottom side and the exposed top side of the cobbles at the modern beach (GUA), since we assume constant movement of the cobbles within the beach berm by wave action; instead constant burial of all cobble sides was assumed for dose rate calculation. All infinite matrix dose rates were calculated with the DRAC software version 1.2 (Durcan et al., 2015).

The contribution of beta radiation from each cobble and its surrounding sediment to the total beta dose rate of individual rock slices used for D_e determination ($\dot{D}\beta_{total}$) were modelled using the approach presented by Freiesleben et al. (2015):

$$\dot{D}\beta_{total} = \dot{D}\beta_{\text{cobble}}\left[1 - 0.5\left(e^{-bx} + e^{-b(h-x)}\right)\right] + \dot{D}\beta_{sed}0.5\left(e^{-bx} + e^{-b(h-x)}\right) \qquad (1)$$

where $\dot{D}\beta_{\text{cobble}}$ (Gy/ka) and $\dot{D}\beta_{sed}$ (Gy/ka) are the infinite-matrix beta dose rates of the cobble and the surrounding sediment, x (mm) is the depth of the slice from the cobble surface, h (mm) is the thickness of the cobble, and b (1.89 mm^{-1}) is the beta linear attenuation coefficient in the cobble. A similar approach was used to model the gamma dose rate, changing the attenuation coefficient b for c (0.01 mm^{-1}). Modelled beta and gamma dose rates for individual rock slices are summarized in Table S4.

The luminescence signal-depth data of all cores were normalized to their individual field-saturation plateau represented by the innermost slices, and fitted with the model of Freiesleben et al. (2015), which can model a sequence of exposure and burial events:

$$L_0(x) \xrightarrow{exposure} L_1(\text{x}) \xrightarrow{\text{burial}} L_2(x) \xrightarrow{\text{exposure}} L_3(x) \qquad (2)$$

Where L_0 is the luminescence signal at depth x (mm) below the rock surface at complete dosing after rock formation, L_1 the luminescence signal after sunlight exposure of the cobble at the beach, L_2 the luminescence signal after burial of the cobble in a raised beach or marine terrace, and L_3 the signal in a previously buried and reactivated cobble. The depth-dependent luminescence signals measured in the collected samples, i.e. $L_2(x)$ for buried surfaces and $L_1(x)$ or $L_3(x)$ for exposed surfaces, can be expressed by the equations:

$$L_1(\mathrm{x}) = L_0(x) e^{-\overline{\sigma\varphi_0} t_e e^{-\mu x}} \tag{3}$$

$$L_2(\mathrm{x}) = (L_1(\mathrm{x}) - 1) * e^{-F(x) t_b} + 1 \tag{4}$$

$$L_3(\mathrm{x}) = L_2(x) e^{-\overline{\sigma\varphi_0} t_e e^{-\mu x}} \tag{5}$$

where t_e (s) is the exposure time at the active beach, $\overline{\sigma\varphi_0}$ (s^{-1}) the effective bleaching rate of the luminescence signal at the rock surface (i.e. the product of photo-ionisation cross section, σ, and the light flux at the rock surface, φ_0), μ (mm^{-1}) the light attenuation coefficient of the rock, F the ratio between total dose rate and the sample-dependent electron trap filling rate constant (D_0), and t_b (s) the burial time in the raised beach or marine terrace.

Analytical determination of the exposure time (t_e) was not possible due to missing data on the local effective bleaching rate, $\overline{\sigma\varphi_0}$, which requires known-age surfaces for calibration (e.g. Sohbati et al., 2012). Likewise, determining the time of burial in inactive landforms (t_b) on the basis of model fitting (this can be achieved when combining t_e and $\overline{\sigma\varphi_0}$ to a single variable, cf. Sohbati et al., 2015) is affected by significant fitting uncertainties. However, fitting of signal-depth data with Eq. (4) was used for evaluating the completeness of signal resetting. Calculation of cobble burial ages in this study was based on measured equivalent doses of rock slices that were identified as bleached by the model, divided by their depth-specific dose rates. All ages were corrected for fading using g-values determined according to Auclair et al. (2003) and the approach of Huntley and Lamothe (2001) for samples from the modern beach, or the model of Huntley (2006) in combination with the approach of Kars et al. (2008) for the Pleistocene terrace samples. In addition, fading correction for all cobbles was determined using the field-to-laboratory-saturation ratio ($[L_{nat}/T_{nat}]/[L_{sat}/T_{sat}]$, cf. Rades et al., 2018). For this, signals in response to ~3000 Gy laboratory doses were normalized with test doses of ~500 Gy (L_{sat}/T_{sat}) and compared to the natural saturation plateau (L_{nat}/T_{nat}) of the luminescence-depth profiles.

3.3 ESR and U-series dating of marine molluscs

Prior to any analyses, X-ray powder diffraction (XRD) measurements were performed at the Laboratory for Physical Geography (University of Cologne) on bulk samples from each of the three mollusc shells (VIR 2-1, SEC 4-2, SEC 4-2a) to check their mineral composition and to evaluate their suitability for combined U-series/ESR dating.

ESR equivalent dose (D_E) values were evaluated using the multiple aliquot additive dose (MAAD) method. ESR measurements were carried out at room temperature at the Institute of Geography (University of Cologne) using an ELEXSYS E500 Bruker X-band ESR spectrometer with a high-sensitivity cavity. All aliquots of a given sample were carefully weighted before ESR measurement to the same mass and centred in the cavity using a teflon sample tube holder. The following acquisition parameters were used for each aliquot: 10 scans, 25.3 mW microwave power, 1024 points resolution, 50 G sweep width, 100 kHz modulation frequency, 0.485 G modulation amplitude measurements, 20.48 ms conversion time and 163.84 ms time constant. Measurements were repeated on two different days. ESR intensities were extracted from peak-to-peak amplitude from the ESR signal at g=2.0006 (Grün, 1988; Bahain et al., 1994; Schellmann and Radtke, 1997, 1999).

U-series analyses of the shells were carried out at the Institute of Geology and Mineralogy (University of Cologne). Three to five sub-samples were collected from each mollusc shell to evaluate the spatial homogeneity of the U-series data. These samples were taken from the shells, not from the powder collected for ESR dating. Isotope ratios and concentrations were measured using a Thermo Scientific Neptune MC-ICPMS with a central SEM. We used an Aridus II desolvator system following a standard-sample bracketing procedure. For U, CRM112A reference material was used and for Th, IRMM35 and IRMM36 standards doped with CRM112A were used. Measured U isotope ratios are corrected for mass bias using the known ratio of the ^{233}U-^{236}U double spike. Thorium samples were doped with the CRM112A U standard, using the known ^{238}U/^{235}U ratio for mass bias correction. The 'Faraday Cup-SEM' yield was corrected using the known ^{234}U/^{238}U or ^{230}Th/^{232}Th ratio of the respective bracketing standard. More details can be found in Obert et al. (in prep) and the Supplementary Information.

ESR and U-series data were combined through USESR, a Matlab-based program (Shao et al., 2014) using the US and AU models defined by Grün et al. (1988) and Shao et al. (2012), respectively. Details about sample preparation, equivalent dose and dose rate evaluation, as well as age calculation can be found in the Supplementary Information.

4. Results and interpretation

4.1 Cobble lithology and feldspar crystal size

For each targeted cobble, polished cutting surfaces of cobble fragments or slices (Fig. 4) were used for petrographic analyses at the Institute of Geology and Mineralogy, University of Cologne. Classification of the cobble lithology and determination of the mineral composition was based on visual inspection under a microscope. All cobbles were composed of a variety of different plutonic, volcanic and subvolcanic rocks (Tab. S3, Fig. 4). Most cobbles with a dark (black to dark grey) colour were alkaline to intermediate volcanic (GUA 1-2, VIR 3-1 P1 and P2, VIR 3-2 P1) or alkaline plutonic rocks (SEC 4-3 P2) that were dominated by pyroxene, biotite and plagioclase. These rocks lacked potassium feldspar or quartz. Cobbles with lighter colours (light grey to pink) represented acidic to intermediate plutonic rocks such as granites (GUA 2-2, GUA 3-1, GUA 3-2), diorites (GUA 1-1, GUA 2-1, VIR 3-2 P2) and monzonites (SEC 4-3 P1) that contained varying amounts of potassium feldspar paired with quartz, plagioclase, biotite and sometimes amphibole.

Visual microscope analysis was also used to estimate average crystal sizes of potassium feldspar minerals used for luminescence dating. The average diameter of feldspar crystals in the granites, diorites and the monzonite was estimated to values between 800±200 µm (GUA 3-2), 600±200 µm (GUA 1-1, GUA 2-1, VIR 3-2 P2), and 400±100 µm (GUA 2-2, GUA 3-1, SEC 4-3 P1) (Tab. S3). The estimated crystal size of plagioclase in basaltic and andesitic rocks was 200±100 µm (GUA 1-2, VIR 3-1 P2, SEC 4-3 P2), and 800±200 µm (VIR 3-1 P1, VIR 3-2 P1) (Tab. S3).

4.2 Luminescence properties of the targeted cobbles

Both IRSL signal (Fig. 5a) and post-IR-IRSL$_{225}$ signal (Fig. 5b) shine-down curves in response to ~30 Gy laboratory doses documented very different sensitivities of the dated cobbles. Cobbles with bright IRSL and post-IR-IRSL$_{225}$ signals (i.e. several 1000 counts per 0.8 s) included GUA 1-1, GUA 2-1, GUA 3-2 and SEC 4-3 P1 (shown as blue lines in Figs 5a,b). Cobble GUA 1-2 had IRSL signals that significantly differed from its background level (~1000 counts per 0.8 s), but no recognisable post-IR-IRSL$_{225}$ signal. With less than 200 counts per 0.8 seconds, all other cobbles had relatively insensitive IRSL and post-IR-IRSL$_{225}$ signals (shown as reddish curves in the insets of Figs. 5a,b).

Laboratory residual doses after 24 hours of solar simulator bleaching (Fig. 5c) were between 0.16 Gy and 2.4 Gy for the IRSL signals of all cobbles. The corresponding post-IR-IRSL$_{225}$ residual doses remained at significantly larger levels between 1.6 Gy and 40 Gy. Dose recovery experiments indicated adequate reproducibility of laboratory-induced IRSL signals for all cobbles with and without residual dose correction (those not corrected for residuals are shown as blue squares in Fig. 5d). The only exception was cobble VIR 3-2 P1, for which extremely dim IRSL signals did not allow for the construction of a dose response curve (Fig. S1). In contrast, the reproducibility of laboratory doses based on post-IR-IRSL$_{225}$ signals was comparatively poor (circles in Fig. 5d). Since residual doses were significant compared to the laboratory doses of ~30 Gy used in the dose recovery experiment, they were subtracted from the recovered doses (triangles in Fig. 5d). However, most samples still yielded inadequate dose recovery ratios. Exceptions were cobbles GUA 1-1, GUA 1-2, GUA 2-1, GUA 3-2 and SEC 4-3 P1, which provided satisfactory dose recovery ratios between 0.9 and 1.1 (blue circles and triangles in Fig. 5d).

Indicators for anomalous fading of feldspar luminescence signals in form of g-values and ratios between signals in field saturation and laboratory saturation ($[L_{nat}/T_{nat}]/[L_{sat}/T_{sat}]$) are shown in Figure S2. Due to comparably dim IRSL and post-IR-IRSL$_{225}$ signals, the g-values of half of the cobbles showed extremely large uncertainties. Only cobbles GUA 1-1, GUA 2-1, GUA 2-2, GUA 3-2, VIR 3-1 P2 and SEC 4-3 P1 (blue symbols in Fig. S2a) yielded g-values with adequate precision in the range of 2.1-4.3 %/decade for IRSL signals (Tab. 1) and 0.4-2.1 %/decade for post-IR-IRSL$_{225}$ signals (Tab. S6). These values are comparable to those reported by Bartz et al. (2020a) for surrounding sand samples using the same measurement protocol. Due to dim luminescence signals in some cobbles the $(L_{nat}/T_{nat})/(L_{sat}/T_{sat})$ ratios varied between less than 0.1 and 0.8 for both the IRSL and the post-IR-IRSL$_{225}$ signal (Fig. S2b). This variability was reduced to ratios of 0.3-0.8 when focussing on cobbles with bright feldspar signals (blue symbols in Fig. S2b), but the ratios still showed no systematic relationship between IRSL and the typically less fading post-IR-IRSL$_{225}$ signals. While ages corrected for fading using g-values and the ratio between field and laboratory saturation differ particularly for cobbles with poor luminescence properties, they agree reasonably well for cobbles with bright signals (Tab. 1, Tab. S6).

4.3 Model fitting of signal-depth profiles

The IRSL and post-IR-IRSL$_{225}$ signal-depth data of cobbles from the modern beach at GUA are presented in Figure 6, those of cobbles from the marine terraces at VIR and SEC in Figure 7. While only data from the shielded surfaces are presented for cobbles from the marine terraces, one core each from the exposed top and the shielded bottom surface were combined to a composite profile in case of the modern beach cobbles. Despite merging both cores in the figure, the signal-depth profiles from each cobble side were fitted individually with Equations (3) or (5). Also, μ values were fitted independently for each surface to allow for small-scale lithological variations within the cobbles. Depth-dependent dose rates were retrieved from Eq. (1) by using the dose rate input data summarized in Table S4. Constant D_0 values for each cobble (Tab. 1, Tab. S6) were estimated by fitting the dose response curves of slices used for conventional equivalent dose determination with a single saturating exponential function. Since the cobbles from the active beach were overturned regularly by waves until sampled for dating, the last event recorded at each surface was assumed to be an exposure event. Remaining signals in the outermost slices, which at the top surface of GUA 2-2 even form a plateau, were interpreted to reflect remnant signals of the ultimate exposure period. For the shielded surfaces of cobbles collected from marine terraces, instead, signal-depth profiles are assumed to end with a burial event. The fitted signal-depth curves of all cobbles are presented in Figures 6 and 7. The associated best-fit model parameters are summarized in Table S5.

Apart from cobble GUA 3-1, for which weak luminescence signals with significant scatter disabled proper fitting of the IRSL signal-depth data, the model fits of all cobbles from the active beach at GUA revealed clear, though varying signs of bleaching at their top and bottom surfaces (Fig. 6). Bleaching of both the IRSL and the post-IR-IRSL$_{225}$ signal similarly affected both sides of the cobbles, without a clear tendency of stronger signal resetting at the bottom or the top surface. However, the IRSL signal was always depleted further into the cobbles compared to the post-IR-IRSL$_{225}$ signal. The latter reflects a single exposure event at the top and bottom surface of all cobbles. The associated bleaching fronts, i.e. the position where signal resetting reaches 50 % of the field saturation plateau in the core of the cobble (cf. Sellwood et al., 2019), remain at shallow depths of 0.1-2.6 mm at both top and bottom surface. Likewise, the measured post-IR-IRSL$_{225}$ remnant signals in the outermost slices of the cobbles remain at high levels of 27-77 % of field saturation at the top and bottom sides of most cobbles (Figs 6b,d,e,f). Only at the top surfaces of GUA 1-1 and GUA 2-1 post-IR-IRSL$_{225}$ signals have been reset to levels below 5 % of field saturation (Figs 6a,c).

Bleaching is generally more effective for the IRSL signal. This is reflected by deeper bleaching front depths of the last exposure event between 0.5 mm (GUA 1-2, bottom surface) and 4.8 mm (GUA 1-1, top surface), as well as smaller remnant signals measured in the outermost slices of 0.7-33 % of field saturation at all cobble surfaces (Fig. 6). Minimum remnant signals of 0.7-1.9 % of field saturation were reached at both the top and bottom side of GUA 1-1 and GUA 2-1. The smaller remnant signals and deeper bleaching fronts also allow the detection of more complex exposure and burial histories for the IRSL signal. According to the fitting, the bottom surface of GUA 1-1 records two exposure events separated by a period of burial (Fig. 6a). The same can be observed at the top surface of GUA 3-2 (Fig. 6f). If the older exposure events recorded in these cobbles are considered, IRSL signal resetting can be proved in even larger depths of up to 6.5 mm, as indicated by the bleaching front of the older exposure event at the bottom surface of GUA 1-1 (Fig. 6a).

In contrast, none of the cobbles collected from marine terraces at VIR and SEC showed unambiguous signs of bleaching and subsequent burial for either the IRSL or post-IR-IRSL$_{225}$ signals (Fig. 7). Although all cobbles except from VIR 3-2 P1 revealed well-defined signal-depth profiles without too much scatter, most of them did not show any indication of bleaching and subsequent dosing and, thus, no fitting was attempted. Only the shielded surface of cobble SEC 4-3 P1 showed slight signs of depleted IRSL and post-IR-IRSL$_{225}$ signals to levels of 57 % and 85 % of field saturation, respectively, in its outermost slice (Fig. 7e). However, fitting of the data did indicate only a single bleaching event and no subsequent burial event.

4.4 Burial dating of cobble surfaces

Extraction of burial ages for Pleistocene terrace cobbles by fitting with Eq. (4) was not possible due to the absence of pronounced bleaching fronts. Thus, just as for the remnant ages remaining in the surfaces of cobbles exposed to sunlight at the active beach (GUA), burial ages recorded in the surfaces of shielded marine terrace cobbles were determined using conventional post-IR-IRSL$_{225}$ burial dose measurements combined with modelled dose rates for each slice. The rock slices used for dating are indicated in Figures 6 and 7, the associated burial doses, dose rates, as well as uncorrected and fading-corrected ages are summarized in Table 1 (IRSL) and Table S6 (post-IR-IRSL$_{225}$).

Bleached sections of the modern beach cobbles yield natural remnant doses as low as 1.2 to 5.0 Gy for IRSL and 2.3 to 18 Gy for post-IR-IRSL$_{225}$, which reduce to 0.1-1.8 Gy (IRSL) and -0.9 to 0.1 Gy (post-IR-IRSL$_{225}$) after subtraction of laboratory residual doses. Fading-corrected remnant ages were than calculated by subtracting fading corrected residual ages from the fading corrected ages of the same sample. This results in fading-corrected remnant ages of 0-800 years for IRSL and -160 to 2600 years for post-IR-IRSL$_{225}$ using g-values and 160-1600 years (IRSL) and 0-7100 years (post-IR-IRSL$_{225}$) using the ratios between field and laboratory saturation (Fig. 8a, Tab. 1, Tab. S6).

The burial doses for surface slices of cobbles from the marine terraces range between 72 and 256 Gy (IRSL) and 96 to >340 Gy (post-IR-IRSL$_{225}$). This equals fading corrected IRSL ages of 36-144 ka using g-values and 40-230 ka using the ratios between field and laboratory saturation. The fading corrected post-IR-IRSL$_{225}$ ages are in field saturation for all cobbles, providing minimum ages between >48 ka and >120 ka (Fig. 8b, Tab. 1, Tab. S6). For the marine terrace cobbles, laboratory residual ages are insignificant compared to their burial ages and were not subtracted. The burial events identified in the IRSL signal-depth profiles of modern beach cobbles GUA 1-1 (bottom) and GUA 3-2 (top) yield residual and fading corrected ages (using g-values) of 1900-2500 years (slice 5) and 700-900 years (slice 2), respectively.

4.5 New ESR and uranium-thorium control ages for marine terrace formation

The three mollusc shells are composed of aragonite, recrystallization to calcite has neither been observed by XRD nor ESR (i.e. no Mn^{2+} signal; Low and Zeira, 1972; Inoue et al., 2000), which suggests that all samples are suitable for ESR and U-series dating.

The U concentration in the shells ranges from 0.34-0.65 ppm (Table S7), which is consistent with observations on marine shells from northern Chile by Radtke (1989). Multiple subsampling provides an idea of the spatial heterogeneity of the U-series data within each shell. While uranium concentrations vary between 3.9 % (VIR 2-1) and 9.1 % (SEC 4-2), the variability of apparent U-series ages ranges from 0.8 % (SEC 4-2) to 7.1 % (SEC 4-2a). These results show a non-negligible spatial variability. All $^{230}Th/^{232}Th$ ratios are >16,000 (Table S7), indicating that there is no detrital thorium contamination in the samples. All analyses return finite U-series age estimates, which suggests that there is no extensive uranium leaching (this was also supported by the combined ESR and U-series data). VIR 2-1 yields a mean U-series age of 128±5 ka, whereas the other two samples provide significantly older ages of 199±1.5 ka and 208±15 ka. All samples yielded initial $^{234}U/^{238}U$ activity ratios (Table S7) that exceed the marine value (1.15), indicating open-system behaviour. Consequently, apparent U-series ages should be regarded as minimum age constraints in first instance, provided that the combination of ESR and U-series data confirms the absence of uranium leaching.

Typical ESR spectra are shown in Figure S3. ESR measurement precision achieved for the three ESR samples is excellent, with a variation of the ESR intensities <2 %. The D_e repeatability over the two different days of measurement resulted in 9.2 % (VIR 2-1), 0.3 % (SEC 4-2), and 5.0 % (SEC 4-2a) (Table S8). A detailed description of the ESR fitting results is provided in the Supplementary Information. Combined U-series/ESR age calculations using the US model do not return any finite age results. Instead, for all samples EU-ESR (EU = Early Uptake; this model is based on the closed-system assumption) estimates are younger than the corresponding U-series ages (Table S9). This demonstrates that the three molluscs experienced uranium leaching (e.g. Grün et al., 1988; Rink et al., 2001). Consequently, only the Accelerating Uptake (AU) model by Shao et al.

(2012) could be used. Sample VIR 2-1 returns an extrapolated AU-ESR age of 88±19 ka. The large age uncertainty results from the massive uranium leaching (ca. 800 %) that has been modelled in order to obtain a finite age result. Samples SEC 4-2 and SEC 4-2a yield younger but consistent AU-ESR age estimates of 56±10 ka and 64±16 ka, respectively. Similarly, the large age uncertainties result from the uranium loss that has been modelled, although much lower compared to that of sample VIR 2-1 (<300 %). Given the non-negligible uncertainty around the modelled uranium leaching, these AU-ESR ages should only be considered as rough approximations. Moreover, although U-series analyses could provide finites age estimates for the three samples, their combination with ESR data shows that the apparent U-series estimates are most likely overestimated as the result of uranium leaching. Therefore, they cannot not be even regarded as minimum age constraints for the molluscs.

5. Discussion

5.1 The significance of cobble lithology for IRSL rock surface dating

As for luminescence dating of sandy deposits, lithology has a crucial influence on the intensity and reproducibility of rock surface luminescence signals. Six out of twelve cobbles in this study revealed dim luminescence signals. Apart from two samples (GUA 3-1, VIR 3-2 P1), residual doses and equivalent doses did provide reproducible equivalent doses and signal depth profiles without significant scatter, although all these cobbles were affected by large uncertainties on their g-values. They could be used to calculate burial ages, which should, however, be interpreted with great care.

Feldspar crystals completely insensitive to IRSL stimulation were also observed by Sohbati et al. (2011) for one out of five magmatic and metamorphic beach cobbles from Denmark, but without further details regarding their specific lithology. In this study, cobbles with dim signals are mostly dark coloured (VIR 3-1 P1 and P2, VIR 3-2 P1, SEC 4-3 P2) and their rather poor behaviour might have been expected due to a lack of significant amounts of potassium feldspar, which is the dosimeter of choice in the post-IR-IRSL$_{225}$ protocol applied in this study. However, also a granite (GUA 3-1) and a diorite cobble (VIR 3-2 P2), both light coloured and containing macroscopic potassium feldspar crystals, revealed dim luminescence signals unfavourable for dating. Cobbles with bright IRSL signals and properties more favourable for luminescence dating in this study are mainly restricted to dioritic cobbles (diorites of GUA 1-1 and 2-1) and granitic rocks (granite of GUA 3-2 and Monzonite of SEC 4-3 P1). With GUA 1-2, a basaltic pegmatite, only one of the intermediate to alkaline magmatic rocks yielded sensitive feldspar.

These more favourable properties for luminescence dating apply to both the IRSL and the post-IR-IRSL$_{225}$ signal. However, for all cobbles, independent of their luminescence characteristics, IRSL signals are at an advantage compared to post-IR-IRSL$_{225}$ signals in terms of intensity, reproducibility and the size of laboratory residual doses. Inadequate post-IR-IRSL$_{180}$ signals for rock samples with IRSL signals suitable for dating were already reported by Souza et al. (2019). The authors suggest that rising signals with illumination time due to isothermal thermoluminescence may be an indicator, and for some samples with inadequate post-IR-IRSL$_{225}$ properties in this study (e.g. VIR 3-1 P1 or VIR 3-2 P2) the same behaviour was observed during isothermal holding prior to stimulation.

Besides controlling the luminescence properties, lithology has a known influence on luminescence signal resetting at rock surfaces (e.g. Ou et al., 2018). Due to different bleaching sensitivities, the resetting of post-IR-IRSL$_{225}$ signals in the cobbles is generally reduced compared to that of IRSL signals. Also, light penetration and signal resetting are much less effective in dark basaltic and gabbroic cobbles (e.g. GUA 1-2), compared to the bright lithologies of some granites and diorites (e.g. GUA 1-1). These differences in IRSL and post-IR-IRSL$_{225}$ signal resetting between light and dark rocks were already described for fluvial cobbles with varying transport distances (Liu et al., 2019) and during laboratory bleaching experiments (Ou et al., 2018).

5.2 IRSL signal resetting in modern beach cobbles

In accordance with observations by Sohbati et al. (2011) and Souza et al. (2019), pronounced bleaching fronts of the IRSL signal were identified in all modern beach cobbles with adequate signal properties (i.e. cobbles with signal-depth profiles not blurred by scatter). The IRSL bleaching front reached maximum depths of 6.5 mm into the rock surface in case of cobbles with light lithology, while darker cobbles reveal less developed bleaching fronts that reach depths of less than 2 mm for approximately the same exposure duration. These bleaching fronts do not show systematic differences between the top and bottom sides of the cobbles at the time of sample collection, which indicates frequent overturning of the cobbles by wave swash and regular changes of the exposed and shielded surfaces. While this was also observed for cobbles from an active beach in Denmark (Souza et al., 2019), cobbles in fluvial systems show a trend towards better bleaching of their upper sides (Liu et al., 2019).

In cobbles with comparably shallow bleaching fronts, surface slices reveal natural IRSL remnants exceeding 8 Gy or 4.8 ka (Fig. 8a). Despite much more pronounced IRSL bleaching fronts that indicate complete signal resetting (e.g. bottom surface of GUA 1-1 or top surface of GUA 2-2, Figs 6a,d), minimum remnant doses of 1.1 to 5.0 Gy, which are equal to ages of 240-1000 years, were measured in their outermost slices (Fig. 8a). This discrepancy may be explained by laboratory residual doses of up to ~2.4 Gy. Subtraction of these laboratory residual doses reduced the inheritance of the surface slices to less than 1.8 Gy or less than 800 years, and to values close to zero for the surface slices of some cobbles. Sohbati et al. (2011) reported similar IRSL laboratory residuals of 2-4 Gy depending on the preheat temperature, but natural remnant doses of only 0.17±0.02 Gy or ~40 years in cobbles from an active beach. Likewise, Souza et al. (2019) reported small IRSL remnant ages of only 130 years for the surface slices of their beach cobbles. The quartz luminescence signals in surface slices of modern beach cobbles from Antarctica even appeared to be completely zeroed (Simms et al., 2011). These discrepancies are likely due to different measurement protocols applied in these studies. As shown by the data of Sohbati et al. (2011), lower preheat and measurement temperatures compared to our post-IR-IRSL$_{225}$ protocol (which was selected since assumed to be more suitable for cobbles with MIS 5 ages) are expected to result in smaller remnant doses.

Comparable to the results of Sohbati et al. (2015) and Ou et al. (2018), the post-IR-IRSL signals of the beach cobbles in this study are reset to a lesser extent than the IRSL signals. The associated bleaching fronts only reach depths of 2.6 mm or less. In consequence, the remnant doses and ages for the most bleached parts of the cobbles are significantly larger. Although laboratory residual doses are larger, as well, and subtraction can reduce the natural remnant doses and ages to less than 0.1 Gy or 160 years for some cobbles, the post-IR-IRSL remnants in the surface slices of most modern cobbles still exceed ~12 Gy or 2400 years after residual subtraction.

5.3 IRSL rock surface dating of raised beaches and marine terraces

Most of the Pleistocene terrace cobbles used in this study revealed unfavourable luminescence properties for dating. Five out of six cobbles have either weak signals, are relatively dark and therefore assumed to allow limited light penetration or both. The granitic cobble SEC 4-3 P1 shows much better luminescence characteristics, which is also the only cobble with IRSL and post-IR-IRSL$_{225}$ signal-depth profiles indicating signal resetting prior to burial (Fig. 7). Fitting is only possible with a single exposure event and, thus, evaluation of signal resetting prior to burial in the terrace body (i.e. through their L_1/L_2 ratios; cf. al Khasawneh et al., 2019) is not possible. The well-developed bleaching fronts identified in modern beach cobbles, however, suggest that in the outermost millimetres at least the IRSL signal was likely reset when the cobble was part of active littoral dynamics. The corresponding residual corrected IRSL remnant doses and ages identified in modern cobbles are insignificant for Pleistocene terrace cobbles from VIR and SEC with burial doses >72 Gy. Resetting of the post-IR-IRSL$_{225}$ signal to sufficiently low levels prior to burial, on the other hand, remains questionable since bleaching to sufficiently low levels (i.e. <5 % of field saturation) is missing in most of our modern analogue samples. Residual corrected remnant doses of 12-32 Gy in the best bleached parts of most modern cobbles are significant even for the presumably MIS 5 marine terrace cobbles investigated here, yielding up to 30 % of the burial dose. Therefore, only IRSL ages that have been corrected for fading using g-values are discussed from here on.

The uppermost slice of SEC 4-3 P1 provides a fading corrected IRSL age of >56 ka years (g-values) and 40±6 ka (ratio between field and laboratory saturation). Disregarding the significant differences between both fading correction approaches – this has been observed in other studies as well, and it remains unclear which approach provides the more robust results for different lithologies (Rades et al., 2018; Ageby et al., 2021) – these cobble ages cannot reproduce the at least MIS 5e age indicated by the post-IR-IRSL minimum age of 110 ka from the sandy beach unit above (Bartz et al., 2020a). An age older than the 40-60 ka cobble ages is also supported by the new ESR/U-series ages on molluscs from the overlying shell layer that were dated to 56±10 and 64±16 ka. The much older U-series age estimates of these molluscs compared to those of ESR dating are regarded unreliable due to uranium leaching (Table S9). This age pattern has also been reported in other ESR dating studies of molluscs from the Chilean coast (Radtke, 1989; Schellmann and Radtke, 1997), and is likely caused by uncertainties due to the open-system behaviour of the molluscs (Radtke, 1989) with associated uranium leaching. As the associated high-energy event deposits can also be found on top of the lower marine terrace at ~12 m a.s.l., which was post-IR-IRSL$_{225}$ dated to MIS 5c and covered by alluvial fan deposits with post-IR-IRSL and quartz ESR ages of ~60 ka (Bartz et al., 2020a), it was likely deposited during MIS 5a and provides a minimum age for the SEC4-3 cobbles (Fig. 8b).

The explanation for the apparent mismatch between cobble ages and age control is most likely field saturation of the IRSL signal. Based on the two times D_0 criterion of the unfaded dose response curve (King et al. 2019a), the IRSL signal of SEC 4-3 P1 has a cobble-specific dating limit of ~340 Gy or 56 ka. Thus, the fading corrected ages of the outermost slice are already in or at least very close to field saturation and can only be interpreted as minimum ages. In addition to that, assuming homogeneous lithology for all cobbles despite partly very different compositions may lead to over- or underestimated dose rates for the surrounding sediments. Although the dose rate of cobble SEC 4-3 P1 is dominated by the high dose rate of the cobble itself (the sediment dose rate contributes only 12-23% to the total dose rate), inaccurate dose rates for the surrounding sediments may contribute to age underestimation as well. The surface age of the second cobble from this unit, SEC 4-3 P2 (74±64 ka), is assumed to be unreliable due to its poor luminescence properties, as also indicated by its extremely large dating uncertainties.

At VIR, fading corrected IRSL ages of cobble surface slices point to ages of >69 ka (VIR 3-2 P2), 24-48 ka (VIR 3-1 P1), and 120-160 ka (VIR 3-1 P2). Similar to those at SEC, these ages cannot confirm the MIS 5c/e age implied by the control ages that indicate deposition between ~90 ka (new ESR/U-series ages and post-IR-IRSL sediment ages from overlying beach sand) and 120-130 ka (post-IR-IRSL sediment age from shoreface sand below) (Fig. 8b). While poor luminescence properties limit the reliability of the rock surface ages for all three cobbles, an additional reason for the overestimated ages of VIR 3-1 P2 may be its dark colour. VIR 3-2 P2 is apparently in field saturation and only provides a minimum age.

Although cobble dating of Pleistocene marine terraces in this study was limited due to inadequate luminescence signals, dark cobble lithologies and/or saturated IRSL signals, good agreement between age control and rock surface ages of Holocene to late Pleistocene beach cobbles were reported from Antarctica using quartz (Simms et al., 2011; Simkins et al., 2013) and from Denmark and Greenland using the IRSL signal of feldspar (Souza et al., 2019, 2021). These studies suggest that the approach has potential for dating littoral cobbles at least on Holocene and late Pleistocene time scales. This is also supported by the burial events identified in two reactivated cobbles from the active beach at GUA. Late Holocene ages of 1900-2500 years and 700-900 years (Tab. 1) agree with relative sea level regression during the past 4000 years following the Holocene maximum (Garrett et al., 2020).

6. Conclusions

Investigation of cobbles from an active beach with ongoing signal resetting by wave motion indicates that IRSL rock surface dating is a promising dating tool for inactive shorelines in Northern Chile. Signals on all sides of the cobbles may be bleached to levels close to zero and feldspar luminescence properties allow for dating Late Holocene burial events. A particular advantage for dating precision is the high cobble dose rates compared to sediment dating, which reduces uncertainties due to water content variations and heterogeneous gamma dosimetry.

However, careful selection of cobble lithology is essential for its successful application. In this study, suitable properties in terms of signal resetting and signal intensity were only associated with granitic and dioritic cobbles with large percentages of bright minerals and significant amounts of macroscopic feldspar crystals. While dark rocks with basaltic and andesitic lithologies that appeared to be unsuitable for dating can easily be excluded during sample collection in the field, our results show that also bright rocks with similar lithology and macroscopic appearance as those suitable for dating, and therefore hard to exclude based on visual inspection, often yield luminescence characteristics inadequate for dating. Additional sources for inaccurate cobble ages are the determination of internal dose rates and fading correction. Both the diameter and potassium content of signal-emitting feldspar crystals in the cobbles were only estimated and previous studies have shown that both parameters may vary significantly between rock types. The approaches applied for fading correction provide partly very different fading rates, while it is still not well understood how to find out the more reliable approach for individual samples. For cobbles from last interglacial terraces, the relatively large cobble dose rates observed in this study imply that IRSL rock surface dating is affected by signal saturation. Dating of such old deposits requires either lower dose rates or higher saturation levels than the cobbles measured in this study, which unfortunately limits the applicability of luminescence rock surface dating to pre-Holocene coastlines to specific lithologies.

Acknowledgements

This study was funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – Projektnummer 268236062 – SFB 1211. Lucas Ageby is funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) - Project number 57444011 - SFB 806. Mathieu Duval’s research is currently funded by the Spanish Ramón y Cajal Fellowship RYC2018-025221-I. We thank María Jesús Alonso Escarza, CENIEH, for technical support with the gamma irradiations. Stefan Opitz (University of Cologne) is gratefully acknowledged for the XRD measurements. We thank Janek Walk (RWTH Aachen University) for his support during field work.

Data availability

The complete dataset of L_x/T_x values of all samples that were used for the fitting of luminescence depth data in this study is available on the database of the collaborative research centre 1211 (DOI: 10.5880/CRC1211DB.42).

Supplementary data

Supplementary material

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Fig. 1: Geomorphological setting of the sampling locations. a) Topographical map of the study area with locations of the sampling sites SEC, GUA and VIR (DEM based on SRTM data). Geomorphological settings and sampling locations of sites SEC (b, c), GUA (d, e) and VIR (f, g) (satellite images in b, d and f © Google Earth 2020).

Figure 2: Stratigraphy of sampled marine terraces at SEC (a) and VIR (b) including the positions of samples for luminescence rock surface dating of cobbles (red, this study), combines U-series/ESR and U-series dating of molluscs (green, this study) and conventional post-IR-IRSL$_{225}$ dating of sand grains (yellow, Bartz et al., 2020a).

Figure 3: Sampling and measurement strategy applied in this study to date beach cobbles with luminescence rock surface dating. a) Schematic cross-sections of a sampled beach profile (GUA) and marine terraces (VIR and

SEC). b) Schematic cobble cross-section with the positions of drill cores and indication of dose-rate contributions. c) Schematic drill core cross-section with measurement strategy for the associated rock slices.

Figure 4: Lithology of the targeted cobbles. In addition to the entire cobbles, one representative rock slice (1 cm diameter) is shown for each cobble.

Figure 5: Luminescence properties of the dated cobbles. Shine-down curves of (a) IRSL signals and (b) post-IR-IRSL$_{225}$ signals in response to test doses of ~30 Gy. Samples with signals significantly different from the background are shown as blue curves, those with dim signals in red (see insets for a close up). c) IRSL and post-IR-IRSL$_{225}$ residual doses after 24 hours of solar simulator bleaching. d) IRSL and post-IR-IRSL$_{225}$ dose recovery ratios of all samples. Adequate ratios, i.e. matching 1.0±0.1 within their uncertainties, are shown in blue.

Fig. 6: Signal-depth profiles with fitting curves of the bleaching model for cobbles from the active beach. Black and red dots indicate IRSL and post-IR-IRSL$_{225}$ measurement data, respectively.

Fig. 7: Signal-depth profiles for cobbles from Pleistocene marine terraces. Black and red dots indicate IRSL and post-IR-IRSL$_{225}$ measurement data, respectively. Fitting of the bleaching model was only possible for cobble SEC 4-3 P1. Open circles indicate slices used for burial dose determination.

Fig. 8: IRSL rock surface remnant ages of modern beach cobbles (a) and IRSL burial ages of Pleistocene terrace cobbles compared to control ages presented in this study (U-series and combined U-series/ESR on molluscs) and previous work (post-IR-IRSL$_{225}$ on sediment, Bartz et al., 2020a). Shaded IRSL rock surface ages in (b) indicate cobbles with unfavourable feldspar luminescence properties. SF – Shore face.

Tab. 1: IRSL burial dating parameters of all targeted cobbles. Cobbles that provide robust ages are marked by shading. Age$_{cor1}$ – fading corrected age using g-values, Age$_{cor1}$-Res – associated age with residual age subtraction, Age$_{cor2}$ – fading corrected age using ratios between field and laboratory saturation, Age$_{cor2}$-Res – associated age with residual age subtraction. *Minimum age estimates based on palaeodoses equal to 2 x D_0.

Lab-ID	Cobble	Core	Depth (mm)	Dose rate (Gy/ka)	D_0 (Gy)	Burial Dose (Gy)	Age$_{faded}$ (ka)	g-value (%/dec)	g-value$_{mean}$ (%/dec)	Age$_{cor1}$ (ka)	Age$_{cor1}$ – Res (ka)	L_{nat}/L_{sat}	$L_{nat}/L_{sat\ mean}$	Age$_{cor2}$ (ka)	Age$_{cor2}$ - Res (ka)
C-L4989	GUA 1-1	GUA 1-1 Top	0-1	6.91±0.73	160	14.2±0.3	2.1±0.2	3.8±1.3	4.3±0.2	3.0±0.4	2.9±0.4	0.44±0.02	0.40±0.02	5.2±0.6	5.0±0.6
			1-2	7.27±0.73		1.4±0.1	0.2±0	4.7±1.3		0.2±0.1	0.1±0.1	0.34±0.02		0.4±0.1	0.2±0.1
		GUA 1-1 Base	1-2	7.27±0.73		2.2±0.1	0.3±0.1	4.5±1.4		0.5±0.1	0.3±0.1	0.40±0.02		0.8±0.2	0.6±0.2
			2-3	7.33±0.73		4.8±0.3	0.6±0.1	4.4±1.5		0.9±0.2	0.7±0.2	0.43±0.03		1.6±0.2	1.4±0.2
			4-5	7.33±0.73		12.3±0.3	1.7±0.2	4.3±1.2		2.4±0.3	2.2±0.[illegible]	-		4.2±0.6	4.1±0.6
	GUA 1-2	GUA 1-2 Top	0-1	2.85±0.34	90	10.2±0.5	3.6±0.6	1.4±1.6	2.7±1.3	4.5±1.[illegible]	4.3±1.1	0.54±0.03	0.49±0.05	7.4±1.1	7.1±1.1
		GUA 1-2 Base	0-1	2.85±0.34		18±0.7	6.3±1	4.1±1.7		8.0±1.9	7.8±1.9	0.44±0.03		12.9±2.2	12.6±2.2
C-L4990	GUA 2-1	GUA 2-1 Top	0-1	4.56±0.34	115	3±0.1	0.6±0.1	1.8±1.1	2.[illegible]±0.2	0.7±0.1	0.5±0.1	0.59±0.02	0.60±0.02	1.1±0.2	0.8±0.2
			1-2	4.79±0.33		1.2±0.1	0.2±0.1	2.1±1.1		0.2±0.1	0±0.1	0.56±0.02		0.4±0.1	0.2±0.1
		GUA 2-1 Base	0-1	4.56±0.34		1.1±0.1	0.2±0.1	2.4±1.1		0.2±0.1	0±0.1	0.64±0.03		0.4±0.1	0.2±0.1
	GUA 2-2	GUA 2-2 Top	0-1	5.86±0.49	60	3.4±0.2	0.6±0.1	2.7±1.9	3.4±0.7	0.7±0.1	0.3±0.1	0.25±0.03	0.28±0.01	2.0±0.3	1.6±0.3
			1-2	6.11±0.49		3.4±0.2	0.6±0.1	2.7±1.9		0.7±0.1	0.3±0.1	0.27±0.03		2.0±0.3	1.6±0.3
			2-3	6.15±0.49		3.7±0.2	0.6±0.1	5.9±2.1		0.7±0.1	0.3±0.1	0.31±0.04		2.0±0.3	1.6±0.3
		GUA 2-2 Base	0-1	5.86±0.49		1[illegible].9±1	2.7±0.4	2.0±1.9		3.7±0.6	3.3±0.6	0.29±0.04		9.7±1.4	9.3±1.4
			1-2	6.11±0.49		[illegible].6±0.5	1±0.2	3.6±2.0		1.4±0.2	1.0±0.2	0.29±0.04		3.7±0.6	3.3±0.6
C-L4991	GUA 3-1	GUA 3-1 Top	1-2	6.1[illegible]±0.4[illegible]	200	96.8±8	15.8±2.6	-0.3±10.5	3.8±9.1	-	-	0.58±0.16	0.41±0.06	38.4±6.2	-
		GUA 3-1 Base	0-1	[illegible].94±0.49		165.6±10.4	27.8±4.1	6.7±2.9		-	-	0.43±0.05		67.9±9.9	-
			1-2	6.15±0.49		56±5.6	9.1±1.6	26.5±20.0		-	-	0.30±0.27		22.2±3.9	-
			2-3	6.19±0.49		81.6±10.4	13.2±2.7	-17.7±6.8		-	-	0.32±0.20		32.2±6.6	-
	GUA 3-2	GUA 3-2 Top	0-1	6.51±0.78	145	2.8±0.2	0.4±0.1	3.6±1.2	3.5±0.4	0.6±0.1	0.4±0.1	0.60±0.03	0.58±0.04	0.7±0.2	0.7±0.2
			1-2	6.65±0.78		5±0.2	0.7±0.1	4.3±1.1		1±0.1.0	0.8±0.1	0.50±0.02		1.3±0.2	1.3±0.2
			2-3	6.67±0.78		18±1.3	2.7±0.5	4.1±1.2		3.7±0.7	3.5±0.7	0.51±0.02		4.7±0.8	4.7±0.8
		GUA 3-2 Base	0-1	6.51±0.78		100.8±4	15.5±2.5	4.7±1.6		21.8±3.8	21.6±3.8	0.50±0.03		26.7±4.2	26.7±4.2
			1-2	6.65±0.78		63±5.3	9.4±1.9	1.9±1.6		13.1±2.8	13±2.8	0.68±0.04		16.2±3.3	16.2±3.3
			2-3	6.67±0.78		199.2±8	29.8±4.7	2.6±1.8		42.3±7.0	42.2±7.0	0.69±0.04		51.4±8.2	51.4±8.2
C-L436	VIR 3-1 P1	VIR 3-1 P1 Base	1-2	2.59±0.59	150	93.6±12.8	36.2±13.2	-0.5±3.	-	36.2±13.2	-	0.29±0.06	-	>108*	-

6								2							
	VIR 3-1 P2	VIR 3-1 P2 Base	0-1	3.21±0.39	570	256.8±12.8	80±13.7	2.8±2.4	2.8±2.4	144±21	-	0.35±0.04	0.35±0.04	229±39	-
			1-2	3.42±0.36		248.8±12	72.7±11.2	-		138±19	-	-		208±32	-
C-L4367	VIR 3-2 P1	VIR 3-2 P1 Base	1-2	1.80±0.50	-	-	-	3.1±13.4	-	-	-	0.12±0.18	-	-	-
	VIR 3-2 P2	VIR 3-2 P2 Base	1-2	2.55±0.68	110	98.4±32	38.6±22.8	8.0±8.0	-	>69*	-	0.45±0.07	-	>69*	-
C-L4368	SEC 4-3 P1	SEC 4-3 P1 Base	0-1	6.32±0.61	210	193.6±8.8	30.6±4.3	2.8±1.3	2.9±0.3	>56*	-	0.76±0.03	0.76±0.03	40±5.6	-
			1-2	6.77±0.49		>340*	>56*	3.0±1.2		>56*	-	-		>56*	-
	SEC 4-3 P2	SEC 4-3 P2 Base	1-2	2.21±0.17	230	72±7.2	32.6±5.8	5.5±2.4	-	59±51	-	0.13±0.08	-	>160*	-

Highlights

- Successful evaluation of luminescence rock surface dating for modern beach cobbles from Chile
- Signal-depth profiles for the IRSL and post-IRSL signals in modern beach cobbles of different lithology
- Application of luminescence rock surface dating to last interglacial marine cobbles from Chile
- Comparison of luminescence rock surface ages for marine cobbles with ESR- and U-series ages of molluscs