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Research paper

# Post-IR $IRSL_{290}$ dating of K-rich feldspar sand grains in a wind-dominated system on Sardinia

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The reliability of a post-IR elevated temperature IRSL (290 °C; $pIRIR_{290}$) is tested on wind-blown, sand-sized (180–250 μm) K-rich feldspar grains. The $pIRIR_{290}$ ages were compared with quartz SAR-OSL data, other independent age controls and historical information. Three study areas along the coast of Sardinia (Italy) were selected: the south Alghero coast, the Bue Marino cave (E Sardinia) and the Alghero bay (NW Sardinia). Along the south Alghero coastline a thick dunefield system is widely recognised in the literature to represent the beginning of the last glacial phase (*post* 80 ka). From a single block sand-sized grains for quartz SAR-OSL and K-feldspar pIRIR dating were collected. The natural quartz SAR-OSL sample lies below the saturation limit of the dose response curve ($D_e < 2xD_0$) giving a reliable age of 76 ± 6 ka. The fading-uncorrected $pIRIR_{290}$ age of 73 ± 5 ka is in good agreement with the quartz result. A further test on older samples was carried out on the sedimentary succession at Bue Marino cave, which includes a sandy wind-blown unit, enclosed between two calcareous crusts. U-series dates of crusts constrain the aeolianite formation between ~130 and ~86 ka. The quartz SAR-OSL signals for aeolianite samples lies close to saturation and the resulting ages underestimate the independent age control. Instead, uncorrected $pIRIR_{290}$ ages on K-feldspar extracts point to a formation of the wind-blown unit between ~100 and ~80 ka, in good agreement with the U-series data. The bleachability of the $pIRIR_{290}$ signal was further investigated using samples from a modern coastal barrier system backing the Alghero bay. The dunefield was stabilized by plantation during the 1950s. The quartz SAR-OSL ages span from 2450 ± 170 years to 60 ± 13 years ago, consistent with the known coastal barrier stabilization. The $pIRIR_{290}$ ages indicate an offset up to ~1000 years. We can conclude that the $pIRIR_{290}$ method on sand-sized K-feldspar grains shows great promise for samples at or beyond the quartz OSL age limit but should not be applied to Late Holocene or modern deposits.



## 1. Introduction

Since the discovery of optical dating on quartz (Huntley et al., 1985) and K-rich feldspar (Hütt et al., 1988) there have been many methodological developments. Especially the development of the Single Aliquot Regenerative-dose (SAR) protocol for quartz (Murray and Wintle, 2000), has improved the reliability of quartz optical dating and since then it has been extensively used to establish chronological framework for Quaternary sedimentary successions of both shallow-marine and continental environments (e.g. Spencer and Robinson, 2008; Jacobs, 2008; Lancaster, 2008; Galli et al., 2009; Andreucci et al., 2010; Thiel et al., 2010; Pascucci et al., 2014; Lamothe, 2016).

The range of OSL dating is usually limited by the saturation of the quartz luminescence dose response curve, which typically occurs at ~150 Gy. Wintle and Murray (2006) suggested an upper dating limit for the quartz OSL (fast component dominated samples) signal to ~$2xD_0$ (i.e. 86% of the saturation level of the dose

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response curve). For samples with natural signals approaching saturation of the dose response curves the reliability is not yet proven and results should be interpreted with caution (e.g. Timar-Gabor et al., 2012). There are also published examples that demonstrate as the quartz SAR-OSL dating method underestimates the age of older deposits (e.g. Buylaert et al., 2007; Lowick et al., 2010; Timar et al., 2010).

Feldspar signals are attractive because their dose response curves saturate at much higher dose than quartz OSL. However, feldspar infrared stimulated luminescence (IRSL) signals measured at ambient temperatures are affected by anomalous fading (Spooner, 1994), which causes age underestimations unless an adequate correction is made (Huntley and Lamothe, 2001). It is claimed in Huntley and Lamothe (2001) that this correction method should not be applied to samples older than 20–50 ka. Furthermore, fading corrections are based on untestable assumptions (e.g. constant fading rates; Morthekai et al., 2008). Recently, research effort was made to identify a feldspar signal which shows less fading (Thomsen et al., 2008). Thomsen et al. (2008) found that a more stable IRSL signal from feldspar can be accessed at higher stimulation temperature when it has been preceded by an initial IR stimulation at ambient temperature (so called post-IR IRSL signals, pIRIR). Since then, the novel feldspar dating protocol with a reduced or negligible fading correction have been tested and developed (e.g. Buylaert et al., 2009; Li and Li, 2011). In this paper we have tested the $pIRIR_{290}$ protocol put forward by Thiel et al. (2011) and Buylaert et al. (2012) on Sardinian aeolian sediment. The $pIRIR_{290}$ results are compared with quartz SAR-OSL ages, independent age control (U-Th ages) and historical information.

## 2. Regional setting and stratigraphy

The Island of Sardinia is located in the north-western Mediterranean Sea (Fig. 1A), emplaced by eastward rotation of the European plate during the Oligocene-early Miocene (Carmignani et al., 2001). In association with this rotation normal and transcurrent faults dissected Sardinian pre-Miocene deposits and allowed the formation of a series of half graben that were filled with Miocene continental and marine deposits and calc-alkaline volcanic bodies (Casula et al., 2001). During the early Pliocene widespread volcanism and basin uplift occurred, but north-west Sardinia is considered to have remained tectonically stable since the Late Pliocene (Lambeck et al., 2004; Ferranti et al., 2006).

The Quaternary deposits crop out quasi-continuously along the study areas and are characterized by an alternation of shallow marine and aeolian/colluvial strata spanning in time from Marine Isotopic Stage MIS 8 (ca 275 ka) to MIS 1 (ca 5 ka; Andreucci et al., 2010, 2011; Pascucci et al., 2014).

### 2.1. Study areas

Three study areas have been selected along the Sardinian coasts: south Alghero coast, Bue Marino cave (Cala Gonone, Orosei Gulf; NE Sardinia) and the Alghero bay (NW Sardinia; Fig. 1B).

The south Alghero coast is characterized by small low-cliff-bounded embayments with basal marine terraces backed by 40 m high coastal cliffs oriented almost parallel to the present-day coastline (Fig. 1C, study area 1). The sedimentary succession which rests on the bedrock, is characterized by 3 m-thick basal shallow-marine strata overlain by 20 m-thick aeolianites deposits. The bedrock is composed of mainly Triassic limestone, Oligo-Miocene andesitic pyroclastic deposits and minor Permian red sandy to gravelly bodies (Pascucci et al., 2014).

The area of Bue Marino (study area 2, Fig. 1D) is characterized by relatively high (50–400 m) and steep Jurassic limestone sea-cliffs affected by karst phenomena. The Bue Marino is a coastal cave is partially filled by wind-blown sediment. The sedimentary succession is encased between sheetlike calcite speleothems (flowstones) (Carobene and Pasini, 1980).

The Alghero area is a west, seaward-opened gulf, laterally bordered by Mesozoic cliffs. The embayment is dominated by N-S oriented 5 km long sandy coastal barrier system delimiting a lagoon named Calick (Fig. 1E, study area 3; Manca et al., 2013; Zucca et al., 2014). The dune system cropping out on the study area has a maximum thickness of 10 m and was human stabilized by plantation during the 1950s.

## 3. Methods

A total of nine samples from the three study areas have been collected for luminescence analyses. Moreover, two samples from the calcareous crusts cropping out at the Bue Marine cave were collected for U-series measurements.

### 3.1. Luminescence analyses

Samples were prepared under sub-due red light conditions at the University of Sassari (Italy) to extract pure fraction of quartz and K-rich feldspar grains following standard procedures (Stokes, 1992). The chosen fraction ranges between 180 and 250 μm and were analysed at the Nordic Laboratory for Luminescence Dating (Department of Geoscience, Aarhus University, DTU Risø campus, Denmark). A portion of each sample (~200 g) was used to estimate the water content and natural radioactivity concentrations, using high-resolution gamma spectrometry following the methodology described by Murray et al. (1987). The total dose rates were then calculated and corrected for water content and cosmic ray contribution (Prescott and Hutton, 1994). The required corrections on the dose rates for cementation (lithified samples) were taken into account and calculated following Andreucci et al. (2009). The lifetime average water content (moisture) since deposition is assumed to lie between present-day and saturated values and has been estimated for each sample based on sedimentological and hydrogeological conditions. K-feldspar grains have an internal radioactivity due to the presence of $^{40}K$ in the crystal structure. Hence, for the K-feldspar dose rates, a contribution to the final dose rate from $^{40}K$ was incorporated based on the assumption that the internal K content is 12.5 ± 0.5% (Huntley and Baril, 1997). The individual radionuclide concentrations, moisture in percentage and total dose rates to quartz and K-feldspar are given in Table 1.

All Luminescence measurements were conducted on an automated Risø TL/OSL reader (model DA-20; Bøtter-Jensen et al., 2010). Quartz was stimulated using blue LEDs (470 nm) and the luminescence detected through a U-340 filter. Feldspar IRSL was stimulated using IR LEDS (875 nm) and detection was through the standard blue filter pack (Schott BG-39 + Corning 7–59). For quartz measurements large size (8 mm) aliquots were used. Because of the bright luminescence signals, feldspar measurements were made on small size (2 mm) aliquots. OSL signal from quartz in this region is known to be dominated by a fast component, as indicated by the steep decay curve of the quartz OSL signal (Andreucci et al., 2010; and Fig. 8 of Thiel et al., 2010).

IR depletion test using infrared stimulated luminescence were conducted on pure-quartz extracts to test residual feldspar contamination (Smith et al., 1990; Duller, 2003). These checks proved negative contamination, so the standard SAR protocol was used for quartz equivalent dose ($D_e$) measurements (Murray and Wintle, 2000, 2003). OSL measurements were made at 125 °C for 80 s, after a pre-heating for 10 s at 260 °C for both samples of Bue Marino cave and South Alghero coast. The pre-heat value was

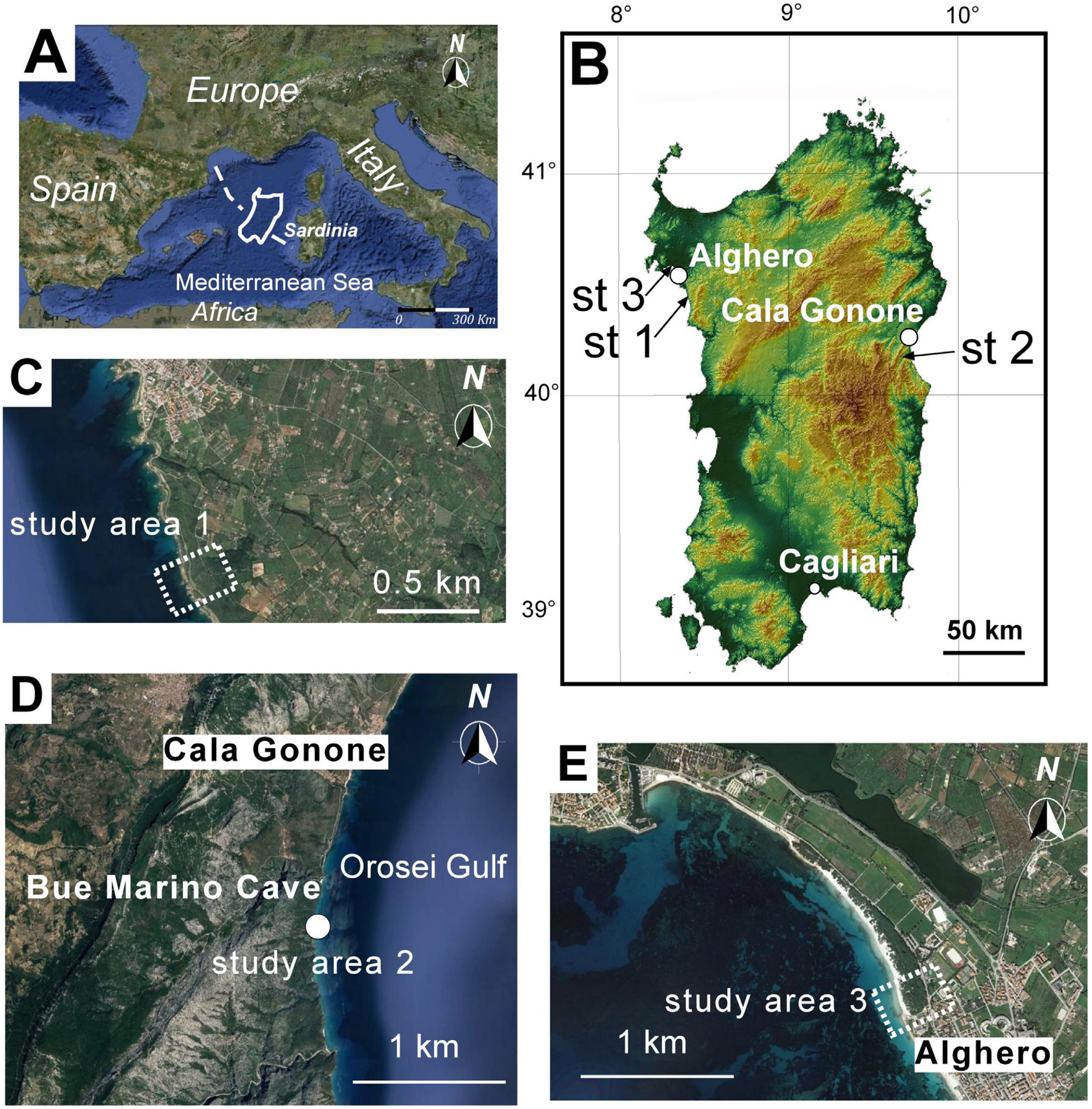


**Fig. 1.** Location map of the study areas. A) Satellite view of the west Mediterranean region. Dashed line indicates the Sardinia rotation occurred in the Neogene time; B) Satellite view of the Sardinia: st1 = South Alghero coast; st2 Bue Marina Cave; st3 Alghero bay. C) Satellite view of the South Alghero rocky coast (study area 1); D) Satellite view of the Orosei Gulf and location of the Bue Marino cave (study area 2). E) Satellite view of the SE side of the Alghero bay and the modern coastal barrier (study area 3). Note the several blow outs, and stabilized by artificial plantation dune system.

experimentally derived on the basis of the results of a dose recovery pre-heat plateau test (data not shown). In this test, we try to mimic in the laboratory, for different experimental conditions, the bleaching and dosing of the samples, a process that has also occurred in nature. A cut-heat of 220 °C was applied to each aliquot before the test dose. A clean out step of a high blue-light stimulation for 40 s at 280 °C was set at the end of each SAR cycle to avoid recuperation of signal. Samples from the modern dune of Alghero bay underwent on lower pre-heat (180 °C) and cut-heat (160 °C) temperatures for the same time to avoid thermal transfer effects (Madsen and Murray, 2009). Five regeneration points were measured during the SAR procedure, including a recycling point repetition of one-step and recuperation (zero given dose step), which were used to determine the effectiveness of the sensitivity corrections of protocol. The initial ~2 s of the luminescence signal, subtracted by the background derived from the 4–8 s, was used for $D_e$ estimation.

A $pIRIR_{290}$ protocol (Thiel et al., 2011; Buylaert et al., 2012) was

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**Table 1**
Summary of principal radionuclides, water contents and total quartz and K-feldspar dose rates for all samples. The quartz and K-feldspar grain size range was 180–250 μm. The conversion factors from activity concentrations to dose rate are taken from Olley et al. (1996). For the K- feldspar dose rates an internal K content of 12.5 ± 0.5% was assumed (Huntley and Baril, 1997).

| Site & location | Sample code | Elevation (m)[a] | $^{238}$U (Bq kg$^{-1}$) | $^{226}$R (Bq kg$^{-1}$) | $^{232}$Th (Bq kg$^{-1}$) | $^{40}$K (Bq kg$^{-1}$) | Moisture[b] % | Quartz dose rate (Gy ka$^{-1}$) | K-feldspar dose rate (Gy ka$^{-1}$) |
|---|---|---|---|---|---|---|---|---|---|
| South Alghero | AHO16 | 2 | 6 ± 4 | 11.7 ± 0.3 | 73 ± 0.3 | 126 ± 4 | 8 | 0.88 ± 0.04 | 1.73 ± 0.08 |
| Bue Marino | BUE1 | 9 | 5 ± 5 | 8.1 ± 0.4 | 16.2 ± 0.5 | 642 ± 13 | 15 | 2.80 ± 0.13 | 3.68 ± 0.14 |
| | BUE2 | 2.5 | 10 ± 6 | 6.5 ± 0.4 | 10.3 ± 0.5 | 580 ± 13 | 5 | 2.20 ± 0.11 | 3.08 ± 0.12 |
| | BUE4 | 6.5 | 10 ± 5 | 9.5 ± 0.4 | 19.0 ± 0.6 | 546 ± 13 | 5 | 2.66 ± 0.12 | 3.54 ± 0.13 |
| | BUE5 | 7.5 | 10 ± 5 | 8.3 ± 0.5 | 11.9 ± 0.4 | 815 ± 16 | 5 | 3.05 ± 0.14 | 3.81 ± 0.15 |
| Alghero Bay | MP4 | 1 | 14 ± 4 | 5.0 ± 0.3 | 8.9 ± 0.3 | 70 ± 3 | 25 | 0.65 ± 0.04 | 1.53 ± 0.07 |
| | MP5 | 3 | 14 ± 3 | 15.3 ± 0.2 | 5.9 ± 0.2 | 93 ± 4 | 25 | 0.69 ± 0.04 | 1.57 ± 0.07 |
| | MP6 | 4 | 10 ± 3 | 11.2 ± 0.2 | 14.3 ± 0.2 | 126 ± 5 | 25 | 0.73 ± 0.04 | 1.61 ± 0.07 |
| | MP7 | 4.5 | 4 ± 3 | 5.4 ± 0.2 | 5.9 ± 0.2 | 226 ± 3 | 25 | 0.73 ± 0.04 | 1.61 ± 0.07 |

Notes:
[a] Elevation corresponds to the elevation of samples collected above the present sea-level.
[b] Moisture corresponds to water content chosen as life-time average for the site. An absolute error of 4% is assumed. The effect of cementation is incorporated in the final dose rate values following Andreucci et al. (2009).

applied to K-rich feldspar grains in order to evaluate the $D_e$ of the same sample measured with quartz SAR-OSL method. After a preheat at 320 °C for 60 s, the samples were bleached with IR diodes at first at 50 °C for 200 s and then again at 290 °C for 200 s to measure the pIRIR$_{290}$ signal. The same preheat conditions were used for natural, regenerative and test dose measurements. An IR illumination at 325 °C for 200 s was inserted at the end of each SAR measurement cycle to minimize recuperation. Fading measurements ($g$ value, Aitken, 1985) were carried out using the same SAR protocol applied for $D_e$ determinations. The $g$ values were normalised to a tc = 2 days (Huntley and Lamothe, 2001). The initial ~2 s of the luminescence signal, less a background derived from the last ~10 s, was used for all calculations. A dose recovery test was also undertaken by adding a dose on top of a very young sample. The quartz OSL and feldspar pIRIR$_{290}$ results for all samples are summarised in Table 2.

### 3.2. U-series method

U/Th extraction from two sheetlike calcite speleothems (flowstones) were carried out at the U-series Laboratory of the University of Bergen (Norway) using an actinide-specific, phosphate/phosphine based (Eichrom TRU) liquid ion exchanger mini-columns, supported on an inert substrate (Amberlite XAD-7) (Hellstrom, 2003; Peterson et al., 2007; Yang, 2009). Isotopic measurements were performed on a Nu Plasma HR multicollector ICP-MS with a U-Pb collector block at the Department of Geology, University of Oslo. Analyses were done in dry plasma using a DSN-100 nebuliser with a sample uptake rate of 0.1 mL/min. The mixed U and Th solution was analysed in two separate procedures. First, uranium isotopes with mass 236, 235 and 234 were determined in ion counters and thorium with mass 232 in a Faraday cup. The second procedure measures thorium mass 229 and 230 in an ion counter. Tailing from 238 to 232 was corrected by measuring half masses and using an exponential interpolation. Fractionation of the instrument has been determined on a daily basis by analysing mass 235 and 238 of a natural uranium solution in Faraday cups using 238/235 = 137.88. The reproducibility of each measured was 0.11% (2σ). Repeated analyses of BR5 (a high uranium speleothem powder standard) gave an age of 125.862 ± 1.546 kyr (n = 12), with a reproducibility of measured $^{234}$U/$^{238}$U ratio of 0.59% (2σ). Ages determinations were based on measured atomic mass ratios of $^{235}$U/$^{236}$U, $^{235}$U/$^{234}$U, $^{236}$U/$^{234}$U, $^{232}$Th/$^{229}$Th and $^{229}$Th/$^{230}$Th. Data reduction, error optimization and propagation were done using tailored software (Lauritzen and Lundberg, 1997), which has been rewritten for the Windows environment. The presence of the non-authigenic $^{230}$Th in the carbonate samples as indicated by the low $^{230}$Th/$^{232}$Th < 20, requires a correction for detrital $^{230}$Th contamination. This correction was performed directly by the software, using initial $^{234}$U/$^{238}$U calculated from $^{230}$Th/$^{234}$U ratio. Analytical results are given in Table 3.

**Table 2**
Summary of quartz OSL and K-feldspar pIRIR$_{290}$ $D_e$ values, pIRIR$_{290}$ fading rates ($g_{2days}$), quartz OSL and uncorrected pIRIR$_{290}$ ages. The number of individual aliquots contributing to $D_e$ is denoted with n. Fading rates are determined on three aliquots previously used for $D_e$ determination. Uncertainties represent one standard error.

| Site & location | Sample code | OSL $D_e$ (Gy) | n | pIRIR$_{290}$ $D_e$ (Gy) | n | $g_{2days}$ (%/decade) | OSL age (ka) | pIRIR$_{290}$ age (ka) |
|---|---|---|---|---|---|---|---|---|
| Alghero bay | MP4 | 0.006 ± 0.01 | 32 | 1.65 ± 0.10 | 3 | 1.05 ± 0.34 | 9 ± 140[b] | 1080 ± 80 [b] |
| | MP5 | 1.69 ± 0.05 | 34 | 4.11 ± 0.10 | 3 | 1.54 ± 0.11 | 2450 ± 170 [b] | 2620 ± 140 [b] |
| | MP6 | 1.22 ± 0.05 | 34 | 3.51 ± 0.12 | 3 | 1.42 ± 0.03 | 1670 ± 100 [b] | 2180 ± 128 [b] |
| | MP7 | 0.040 ± 0.008 | 32 | 2.29 ± 0.16 | 6 | 1.58 ± 0.05 | 60 ± 13 [b] | 1422 ± 120 [b] |
| South Alghero | AHO16 [a] | 67 ± 4 | 30 | 127 ± 7 | 9 | 1.44 ± 0.19 | 76 ± 6 | 73 ± 5 |
| Bue Marino | BUE1 | 141[c] | 4 | 317 ± 7 | 6 | 1.77 ± 0.13 | 50[c] | 86 ± 4 |
| | BUE2 | 168[c] | 4 | 332 ± 6 | 6 | 1.19 ± 0.20 | 76[c] | 108 ± 5 |
| | BUE4 | 151[c] | 4 | 296 ± 10 | 5 | 1.63 ± 0.26 | 57[c] | 84 ± 4 |
| | BUE5 | 165[c] | 4 | 338 ± 9 | 6 | 1.45 ± 0.09 | 54[c] | 89 ± 4 |

Notes:
[a] Data from Buylaert et al. (2012).
[b] Age expressed in years from Andreucci et al. (2010).
[c] The natural signal lies above the 86% of the saturation level of the dose response curve (see Fig. 2b). The dose corresponding to 2*$D_0$ (86% saturation) is given and the quartz age is considered a minimum age.

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**Table 3**
Summary of Uranium concentration, U/Th ratios, row and corrected ages. Uncertainties represent two standard error (2σ).

| ID lab | ID sample | U (ppm) | $^{234}U/^{238}U$ | 2σ+ (%) | 2σ− (%) | $^{230}Th/^{234}U$ | 2σ+ (%) | 2σ− (%) | $^{230}Th/^{232}Th$ | 2σ+ (%) | 2σ− (%) | Age (ka) | Initial $^{234}U/^{238}U$[a] | Corrected age (ka) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 857 | AL3-2 | 0.0257 | 1.035 | 2.79 | 0.03 | 0.647028 | 6.67 | 0.04 | 4 | 6.11 | 0.27 | 113.3 ± 14 | 1.04 ± 0.029 | **86.1 ± 13** |
| 855 | AL2-stm | 0.0280 | 0.958 | 3.99 | 0.04 | 0.702972 | 10.94 | 0.08 | 21 | 10.19 | 2.15 | 139.5 ± 32 | 0.93 ± 0.037 | **134.2 ± 32** |

[a] Data deriving from $^{230}Th/^{234}U$ ratio.

## 4. Results

### 4.1. Study area 1: South Alghero coast

A sample block (ca 50 × 50 × 40 cm) of aeolianite (AHO16) was collected at Alghero coast (Fig. 2A) at the base of the 20 m-thick relic dune system (Fig. 2B). Sampled block is characterized by medium to coarse-grained carbonate-rich sandstone made up of: 90% $CaCO_3$, 8% siliciclastics, 1% fines and 1% organics.

The quartz SAR-OSL $D_e$ value is 67 ± 4 Gy and the quartz dose rate is 0.88 ± 0.04 Gy/ka; both values are typical for Sardinian aeolianites (Andreucci et al., 2009, 2011). The studied sample shows good OSL characteristics. It is dominated by a fast component and it has an acceptable recycling ratio of 1.12 ± 0.14 (n = 12) within 10% uncertainties and the results of the dose recovery test (Dose recovery ratio of 1.02 ± 0.04, n = 18) is satisfactory. Because of the low dose rate, the quartz OSL signal lies below the saturation limit (69% of saturation, data not showed) and we conclude that AHO16 has a reliable quartz age of 76 ± 6 ka. The K-feldspar $pIRIR_{290}$ $D_e$ value is 127 ± 7 Gy and the total dose rate is 1.73 ± 0.07 Gy/ka. The K-feldspar sample has a fading-uncorrected age of 73 ± 5 ka, in good agreement with the quartz OSL age.

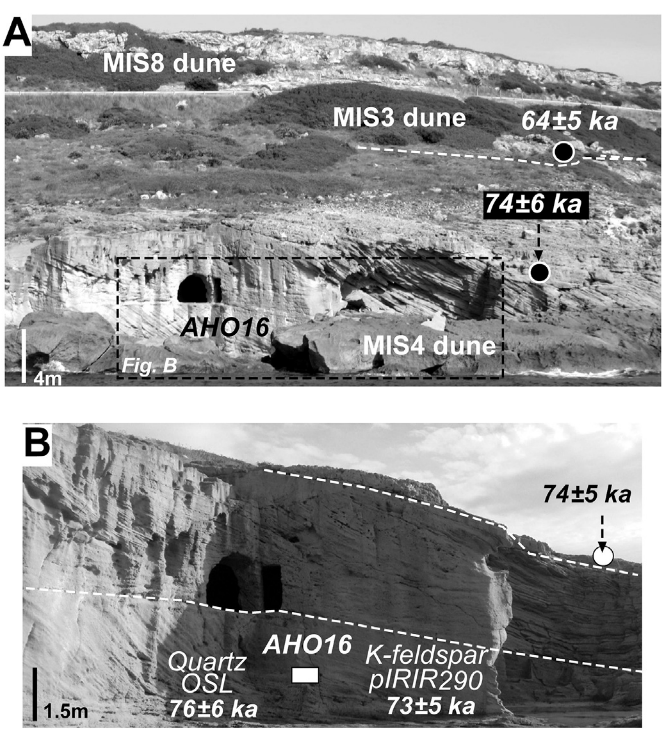


**Fig. 2.** Study area 1: South Alghero coast. A) Panoramic sea view of the 20-m thick transgressive dune body. Note that the aeolian deposits span in time from the Marine Isotopic Stage (MIS 4) to MIS 3. On the top of the hill a MIS 8 (250 ka) dune crops out. The black circles indicate previous OSL ages (modified from Andreucci et al., 2010). B) Detailed view of the basal part of the dune body. The white square indicates the sampling location of the new sample (AHO16) together with the Qz and K-feldspar age for this sample. White dot is the OSL age from Andreucci et al. (2010).

### 4.2. Study area 2: Bue Marino cave

At Bue Marino cave samples were collected along a 7 m-thick yellowish sandy aeolinite scarp capped by a 50 cm-thick reddish sandy colluvial deposits. The succession is bounded at the base and at the top by 50 cm-thick calcareous crusts (flowstones) (Fig. 3). One opaque PVC tube (D = 8 cm; L = 40 cm) was hammered into a freshly exposed reddish colluvial strata (BUE1) (Fig. 3A) Three sample blocks (ca 50 × 50 × 40 cm) of aeolianites (BUE2, BUE4, BUE5, from the base to the top) were collected (Fig. 3A–C; Table 2). Aeolianites are characterized by medium to coarse-grained carbonate-rich sandstone made up of 70–82% $CaCO_3$, 16–28% siliciclastics, 1% fines and 1% organics. Instead, colluvial strata are composed of a matrix-supported sandy to pebbly deposit made up of 31% $CaCO_3$, 13% siliciclastics, 55% fines and 1% organics.

Quartz equivalent dose ($D_e$) values range between 141 and 165 Gy (BUE1 141 ± 5; BUE2 168 ± 8; BUE4 151 ± 6; BUE5 165 ± 9 Gy) increasing from the top to the bottom of the succession. A dose recovery ratio test on sample BUE4 gave a satisfactory measurement of 0.99 ± 0.05 (n = 12). The quartz dose rates range from 2.21 to 3.05 Gy/ka, which is consistent with the dose rates of palaeosols in the area but is relatively high for Sardinian aeolianites (Andreucci et al., 2011). Thus, given such high dose, rate OSL signals for BUE samples are close to saturation ($D_e > 2D_0$) and quartz estimated ages are likely to be only minimum ages ranging between 76 and 50 ka (Table 2).

The $pIRIR_{290}$ K-feldspar $D_e$ values range between 296 ± 10 and 338 ± 9 Gy (BUE1 317 ± 7; BUE2 332 ± 6; BUE4 296 ± 10; BUE5 338 ± 9 Gy) and the K-feldspar dose rates vary between 3.02 and 3.86 Gy/ka. A dose response curve of sample (BUE2, $D_e$ 332 ± 6 Gy) shows that the natural feldspar signal lies far below the saturation level of the curve (Fig. 3D). The dose recovery test for the K-feldspar $pIRIR_{290}$ protocol was carried out by adding a beta dose (49 Gy) on top of a very young K-feldspar sample (MP7; study area 3, see following section) and subtracting the natural $D_e$ (1.9 ± 0.2 Gy) from the measured dose. The dose recovery ratio is 0.97 ± 0.08 (n = 16) confirming the suitability of this protocol for measuring laboratory dose given prior to thermal heat treatment. Fading measurements were carried out on three aliquots from each sample after measuring the $D_e$ values. The mean $pIRIR_{290}$ $g_{2days}$ value is 1.50 ± 0.10%/decade (n = 12). It has been suggested (Buylaert et al., 2012; Roberts, 2012) that these low fading rates may be artefacts of our measurement procedure and that the evidence that these apparent fading rates reflect loss of charge during storage is not convincing. Therefore, we chose not to correct the ages for fading.

The uncorrected $pIRIR_{290}$ ages range from the base to the top: 108 ± 5 ka, 84 ± 4 ka, 89 ± 4 ka and 86 ± 4 ka. U-series dates of the flowtstone (Table 3) that are bounding the sedimentary succession give ages of 134 ± 32 ka at the base and 86 ± 13 ka at the top; we conclude that our uncorrected $pIRIR_{290}$ ages are in good agreement with this independent age control.

### 4.3. Study area 3: Alghero bay

Four opaque PVC tubes (D = 8 cm; L = 40 cm) of freshly exposed modern coastal deposits (MP4, MP5, MP6, MP7) were collected

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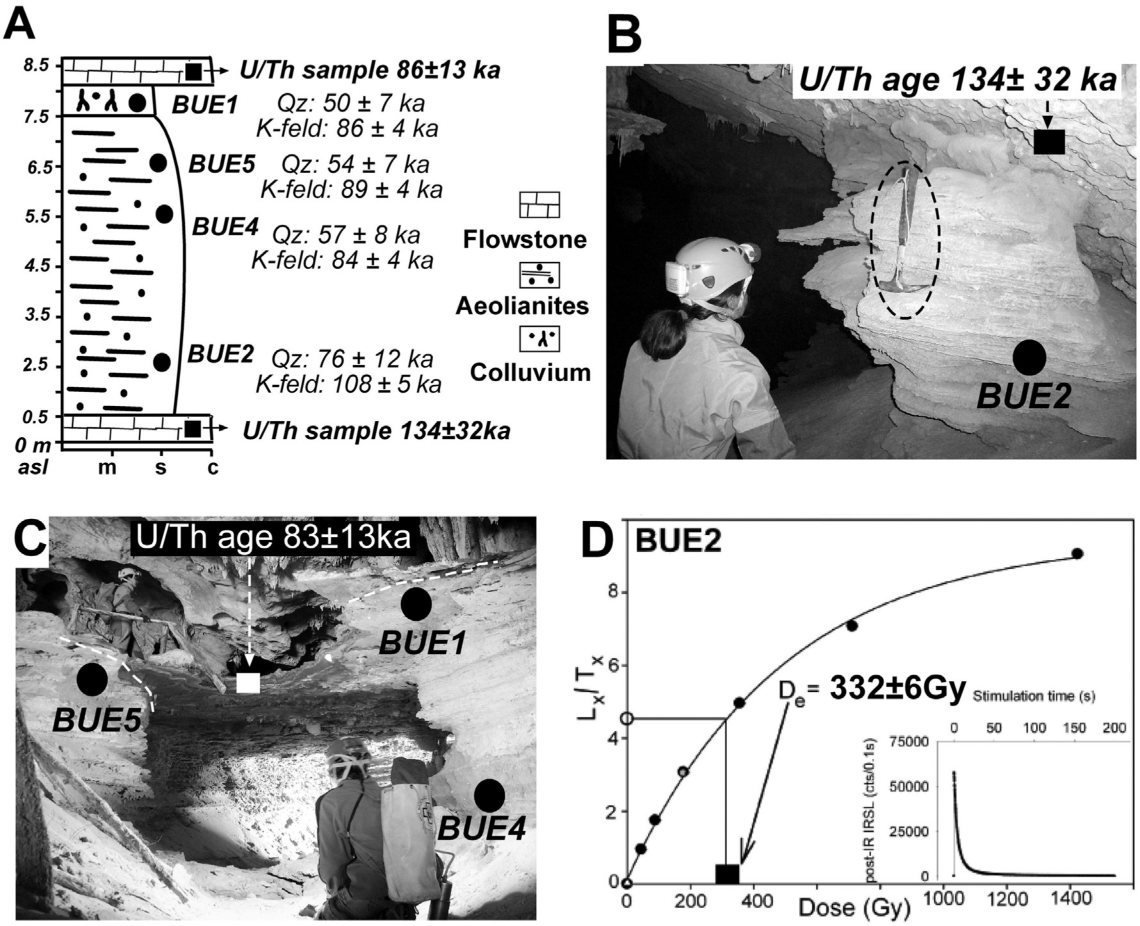


**Fig. 3.** Study area 2: Bue Marino cave. A) Schematic drawing showing the relationship between samples at the Bue Marino cave. B) Detailed view of the basal part of the aeolian deposits. Note that the sedimentary body drapes the cave flank is encrusted by a calcareous crust (flowstone). The black square indicates the sample position for U/Th analysis and in bold letters the U/Th ages. The black circles refer to the luminescence sampling positions. Scale on the left side is a hammer (40 cm high). C) detailed view of central to top parts of the succession characterized, from bottom up, by wind blown sand and thin colluvial strata. The succession is capped by a 50 cm-thick flowstone with a U/Th age of 83 ± 13 ka. Scale on the right side is a sitting woman (1 m high). D) K-feldspar pIRIR$_{290}$ dose response curve for sample BUE2 showing a recycling point (grey circle) and a recuperation point (white triangle). The insets shows the natural pIRIR$_{290}$ decay curve. Note that sensitivity corrected natural signal (white circle) is well below the saturation limit of the growth curve.

(Fig. 4; Table 1). One sample (MP4) was collected on the modern backshore, in front of an active blow out (Fig. 4A), Three samples (MP5, Fig. 4B, and MP6, MP7 Fig. 4C) were collected on a natural 5 m-thick scarp carved into the stabilized present dunefield. Samples are characterized by medium to coarse-grained carbonate-rich sandstone made up of: 55–75% $CaCO_3$, 33–43% siliciclastics, 1% fines and 1% organics. The studied samples shows good OSL characteristics such as recycling values within 5% of unity and a dose recovery ratio with a mean value of 1.03 ± 0.08 (n = 9). Quartz $D_e$ values (MP4 0.006 ± 0.01 Gy; MP7: 0.040 ± 0.008 Gy, MP6: 1.22 ± 0.05 Gy, MP5: 1.69 ± 0.05 Gy) increase significantly from the top to the base of the succession, as expected. The dose rate values of all samples are around ~0.7 Gy/ka as expected for quartz-rich beach-dune deposits of Sardinia (i.e. Andreucci et al., 2010; Pascucci et al., 2014). Samples collected on the natural scarp (MP5, MP6, MP7) from the base to the top have ages of 2450 ± 170 a, 1670 ± 100 a, and 60 ± 13 a respectively. The sample (MP4) from the modern backshore zone has a $D_e$ value of 0.006 ± 0.01 Gy resulting in an age consistent with present day (9 ± 14 years). The pIRIR$_{290}$ ages for this sample is 1080 ± 80 years which is clearly too old. Also for the other samples, the pIRIR$_{290}$ ages are a few hundred to a thousand years older than the quartz OSL ages. It is worthy to note that regression of the Alghero coastal system most probably started about 2500 years ago, corresponding with the Roman Optimum (Mensing et al., 2015).

## 5. Discussion

The thick lithified dune system along the south Alghero coast (study area 1) is widely recognized in the literature to represent the beginning of the last glacial phase (*post* 80 ka; Andreucci et al., 2010; Pascucci et al., 2014; Fig. 2). Because of the low dose rate of this material (<1 Gy/ka), the quartz OSL signal is not in saturation, the $D_e$ is 67 ± 4 Gy and the estimated age of 76 ± 6 ka is considered reliable. The age is also in good agreement with the previous study carried out on this material by Andreucci et al. (2010) and (2011) (Fig. 2). Thus, this sample can be used to test the reliability of feldspar pIRIR$_{290}$ dating on this material (Buylaert et al., 2012); the fading-uncorrected pIRIR$_{290}$ age of 73 ± 5 ka is in very good agreement with the SAR-OSL. For further testing of the pIRIR$_{290}$ dating method we used the older samples with larger equivalent doses, such as can be found in study area 2 (Bue Marino cave).

The results from the Bue Marino cave, where we have independent age control from U-series dating, indicate that the quartz-OSL signal is close to saturation when $D_e$ values of between 141 ± 5 Gy (BUE1) and 165 ± 9 (BUE5) are measured. The corresponding quartz OSL ages for the upper samples are ~50 ka (BUE1) and for the lowermost sample ~80 ka (BUE2) (Fig. 3C), which

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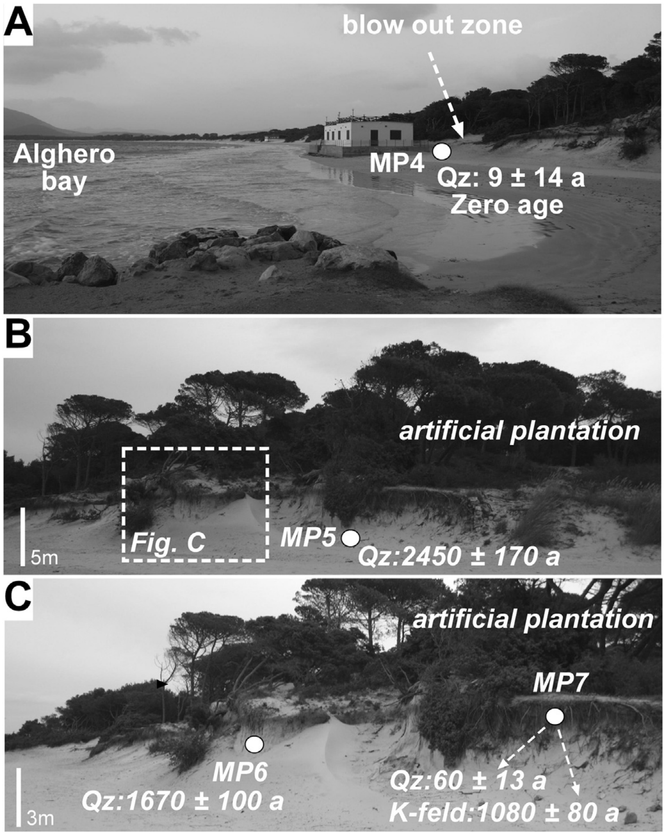


**Fig. 4.** Study area 3: Alghero bay. A) Panoramic view of the Alghero bay, sandy beach system and dunes human stabilized during the 50th. The white circle indicates luminescence sampling positions and the obtained quartz and feldspar ages B) View of the lower part of the dunes. C) Detailed view of the top part of the dune body.

strongly underestimate the constraining age controls. Contrarly, $pIRIR_{290}$ measurements on K-feldspar gave uncorrected ages between ~108 and ~86 ka, in good agreement with the constraining U-series data. Moreover, independent ages constraining gap provided by U-series is too wide to be able to test whether fading correction needs to be carried out for $pIRIR_{290}$.

The bleachability of the $pIRIR_{290}$ signal has been investigated based on the analysis of samples collected in the Alghero modern dune system. It is well known that the quartz fast component can be bleached to negligible levels and be used to date reliably very young samples (Madsen and Murray, 2009). Indeed, it can be seen that modern sample (MP4) gave an age (9 ± 14 a) consistent with zero years (Fig. 4A). The quartz ages of the other three samples are in stratigraphic order (Fig. 4B and C) and range between 2450 ± 170 at the bottom to 60 ± 13 at the top. Given the aeolian nature of the sediments and their good luminescence characteristics, we consider these quartz OSL ages reliable. When comparing the $pIRIR_{290}$ ages with the quartz OSL results, it can be seen that there is an offset of a few hundred to one thousand of years for these samples. The $pIRIR_{290}$ $D_e$ value of 1.65 ± 0.10 Gy (n = 3) for sample MP4 (quartz OSL $D_e$ ~ 0 Gy) may represent the unbleachable residual dose of the $pIRIR_{290}$ signal or it may be the result of thermal transfer during preheating (Buylaert et al., 2011) and thus $pIRIR_{290}$ signal has been well reset. We therefore suggest that whether possible, modern or very young feldspar samples and quartz OSL age control should have been always measured to be able to place a lower limit to the residual $pIRIR_{290}$ doses for investigated sediments. It should also be mentioned that for such young sediments good pIRIR results have been obtained using lower preheat and stimulation temperatures (e.g. Madsen et al., 2011; Reimann et al., 2012).

## 6. Conclusions

This study has contributed to the further testing of the post-IR elevated temperature (290 °C) IRSL protocol ($pIRIR_{290}$) applied to coarse-grained K-feldspar for dating well-bleached sedimentary samples. We conclude that:

1. The dating technique shows great promise for dating coastal deposits in Sardinia that are at or beyond the quartz OSL dating limit (2 × $D_0$), as indicated by the agreement between the uncorrected $pIRIR_{290}$ ages and the bracketing U-Th ages.
2. Because the independent age constraints on our samples is limited, we cannot conclude whether the $pIRIR_{290}$ ages need to be corrected for the small laboratory fading rates of ~1.5%/decade.
3. The $pIRIR_{290}$ protocol shows a significant age offset of a few hundred to a thousand years for young (Holocene) samples but such a residual becomes quickly insignificant as deposits get older.

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