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Toward a luminescence chronology for coastal dune and beach deposits on Calvert Island, British Columbia central coast, Canada



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ABSTRACT

The Quaternary geology of the central coast of British Columbia contains a rich and complex record of glacial activity, post-glacial sea level and landscape change, and early human occupation spanning the last ~10,000 years. At present, however, this region remains a largely understudied portion of coastal North America. This study describes the luminescence characteristics of quartz and K-feldspar from coastal dune and beach sands on Calvert Island and develops a suitable optical dating protocol that will allow for a more rigorous chronology for post-glacial landscape evolution and human occupation on British Columbia's central coast. Luminescence signals from Calvert Island quartz are dim, and appear to lack the so-called "fast" component that is most desirable for optical dating. K-feldspar signals are sufficiently bright for optical dating. We test and refine a single-aliquot regenerative-dose (SAR) protocol for K-feldspar specific to Calvert Island samples through a series of dose recovery and preheat plateau tests. Two approaches for correcting a sample age for anomalous fading are compared and a correction for phototransfer is introduced and applied. Measured fading rates vary from sample to sample implying that, in this region, it is not sufficient to rely on two or three representative fading rates as has sometimes been done elsewhere. Refined age estimates show consistency with independent radiocarbon dating control and help identify radiocarbon-dated organic-rich sediments that have been reworked.

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1. Introduction

Recent research on the central coast of British Columbia (BC), Canada, suggests that it has a unique postglacial history as sea level has remained remarkably stable since deglaciation and archaeological evidence exists for continuous human occupation for at least the last 10,000 years (Shugar et al., 2014; McLaren et al., 2014). Calvert Island, located south of Hakai Pass, hosts a variety of coastal landforms including bluff- and dune-backed embayed beaches, tombolos, and stabilized dune complexes that contain important records of environmental change, landscape evolution, and human occupation during postglacial time. This part of the BC coast is a focus of the Hakai Institute, which supports several collaborative geographical, geological, biological, and archaeological research

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projects in the area. It is, therefore, important that a means of providing a reliable chronology for landscape change in the region be developed and tested.

Radiocarbon ages have provided a temporal framework for coastal landform development and human activity, but a more comprehensive and robust chronology can be achieved by combining them with optical ages, which provide a source of chronological information at sites where datable organic material is not present or where it may be reworked. In the past, optical ages from K-feldspar sediments have been used in palaeoenvironmental studies of BC's coast but until now, methods of determining an equivalent dose (D_e) and anomalous fading rates have been largely confined to multiple aliquot dating techniques (e.g., Huntley et al., 1985, Lian et al., 1995; Huntley and Clague, 1996; Huntley and Lamothe, 2001; Wolfe et al., 2008).

In this study, we examine luminescence signals from both quartz and K-feldspar from Calvert Island to confirm that K-feldspar is the most suitable mineral for optical dating in this region. We

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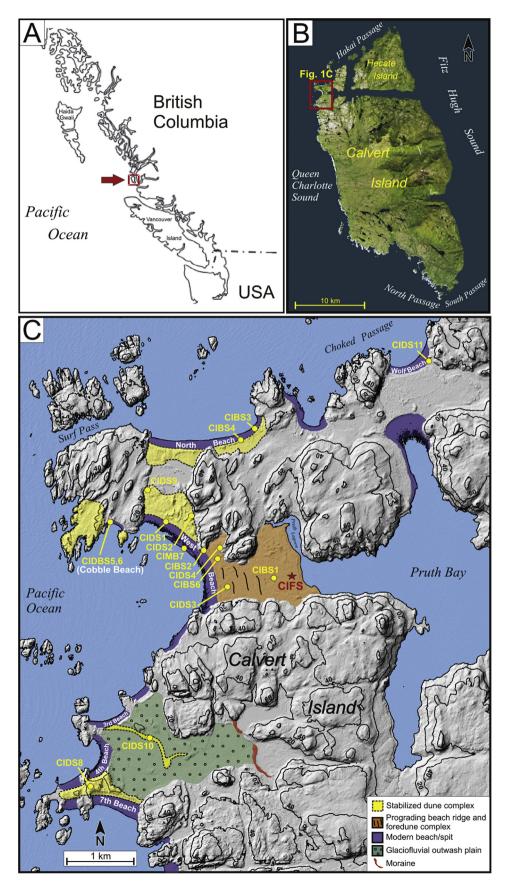


Fig. 1. A) Location of Calvert Island, BC central coast. B) Calvert and Hecate Islands, south of Hakai Pass. C) Prominent glacial features, modern beach deposits, and dune and beach ridge complexes superimposed on a 2 m LiDAR hillshaded DEM of northwest Calvert Island. Optical dating sample sites are shown as yellow dots. CIFS = Calvert Island Field Station of the Hakai Institute. Contour interval is 20 m. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

then test and refine a single-aliquot regenerative dose (SAR) protocol for Calvert Island K-feldspar through a series of dose recovery and preheat plateau tests. Two approaches to correcting a sample for anomalous fading are tested and compared and a new correction procedure for phototransfer (cf. Huntley and Clague, 1996; Ollerhead and Huntley, 2011) is introduced and applied. Age estimates using our refined procedure are compared with associated radiocarbon ages at three sites.

2. Study area

Calvert Island, and its northern neighbour, Hecate Island, are located ~50 km south of Bella Bella (Fig. 1A). They are south of Hakai Passage, a 2–3 km wide ocean corridor that separates northern Hecate Island and Hunter Island (Fig. 1B), and are separated from the mainland Coast Mountain Ranges by Fitz Hugh Sound. The central and eastern parts of Calvert Island are characterized by glacially eroded mountain ridges and peaks reaching up to ~1000 m above sea level (asl). The bedrock is composed mainly of early Cretaceous tonalite, quartz diorite, granite, granodiorite and diorite of the Calvert Island Pluton with diorite-dominated rocks of unknown age cropping out mostly in the central, eastern, and southeastern parts of island (Roddick, 1996). West of the mountain peaks, the terrain slopes down to less than ~200 m asl and is overlain by alluvial and glacial sediments (Roddick, 1996). The northwestern part of Calvert Island consists of bluff-or dunebacked beaches, including two prominent beaches, North Beach and West Beach (Fig. 1C). Large foredune systems backed by stabilized dunes have developed between North and West beaches and on the peninsula between Fourth and Seventh Beaches (Fig. 1C). A large, vegetated, prograding beach ridge and foredune complex lies between Pruth Bay and West Beach. Dunes have also been identified on a late Pleistocene glaciofluvial outwash plain that lies west of two moraines (Fig. 1C).

3. Sample collection and measurement

On northwestern Calvert Island, optical dating samples were collected from recently deposited (i.e., modern) and ancient dune and beach sediments (Fig. 1C, Table S1). Two samples, CIDBS5 and CIDBS6, were collected from a unit that is likely dune sand or beach sand overlying gravel and a peat bed on Cobble Beach. Optical dating samples were collected by hammering aluminium tubes (~7.5 cm diameter) into a cleaned face. The tubes were then extracted and sealed. Where possible, samples for accelerator mass spectrometry (AMS) radiocarbon dating were also collected. See Tables S1 and S2 for sample locations, elevations, and the stratigraphic contexts of all optical and radiocarbon sample sites. See Supplementary Material and Table S3 for details on sample preparation, the SAR procedure, dosimetry measurements and dose rate data.

4. Results

4.1. Calvert Island quartz optically stimulated luminescence signals

Natural continuous-wave optically stimulated luminescence (CW-OSL) signals from Calvert Island quartz are very dim (Fig. S1). Linearly modulated (LM-) OSL curves show that Calvert Island quartz lacks the thermally-stable fast component desirable for optical dating (Fig. S1). It is likely that the quartz sediment in our samples has been eroded from the Calvert Island Pluton and/or other plutonic sources on the mainland by glaciers during the last glaciation, and/or a portion of it has been derived locally by rapid weathering since the Last Glacial Maximum. If so, then the Calvert

Island quartz sediment has likely experienced relatively few cycles of erosion and deposition that allow for repeated bleaching and irradiation, which is thought to increase the sensitivity (and brightness) of its luminescence signal (cf. Pietsch et al., 2008). Thus, all age estimates were obtained from K-feldspar.

4.2. Testing a SAR protocol for K-feldspar

Equivalent dose (D_e) measurements were initially made using a SAR protocol that was shown to be suitable for K-feldspar in the Fraser River valley, south-central BC (unpublished data). This protocol included a 220 °C preheat before measurement of the natural (L_n) and regenerative (L_x) dose signals and a 160 °C preheat before measurement of the test dose (T_x) signal (Table S4). When applied to Calvert Island K-feldspar this protocol yielded recuperated signals ranging from ~6% to ~118% of the natural signal with the highest recuperation appearing in the youngest samples (cf. Li et al., 2006; Madsen and Murray, 2009) (Table S5). Ideally, recuperation rates should remain below ~5% to be acceptable for dating (Murray and Wintle, 2000). Two approaches were taken to try to reduce the recuperation, and these included introducing a so-called "hotwash" into the SAR sequence and adjusting the L_n and L_x preheat temperatures.

4.2.1. Introducing a hotwash

Recuperation and build-up of background signal during a SAR cycle may be minimized by applying a high-temperature optical stimulation at the end of each SAR cycle, otherwise known as a hotwash (Murray and Wintle, 2003). Dose recovery tests were conducted on 24 K-feldspar aliquots from sample CIDS1; 12 with a 40 s hotwash at 180 °C and 12 without a hotwash (Fig. 2A). The hotwash appears to have little effect on the aliquot measured-togiven dose ratios and recuperation values are noticeably lower, most below 5% (Fig. 2A, inset graph). Therefore we've included a hotwash that is at least 20 °C higher than the preheat temperature in all subsequent D_{ℓ} measurements.

4.2.2. Adjusting the preheat

A preheat plateau test was conducted on 21 aliquots from sample CIDS1 that had been bleached in the sun for two days and then given a laboratory dose of ~1 Gy. A range of preheat temperatures (160–280 °C) were applied preceding the L_n and L_x measurements, and of these, 160 °C yielded the best results (Fig. 2B). Recuperation values and recycling ratios were within acceptable limits for all preheat temperatures. Measured-to-given dose ratios increased with increasing preheat temperatures between 220 and 260 °C. An anomalously low measured-to-given dose value was measured at 280 °C; this resulted in a distorted dose response curve (Fig. 2C) and likely reflects thermal degradation of the infrared stimulated luminescence (IRSL) signal at high preheat temperatures. All subsequent D_e measurements have thus been made using a low (160 °C) preheat temperature (SAR protocol 2, Table S4).

4.2.3. Dose recovery tests and phototransfer effects

Dose recovery tests were performed on two samples of different age (CIBS3 and CIDS1) to test the suitability of Calvert Island K-feldspar from both young (<200 years) and older (>1000 years) samples to our refined SAR procedure (protocol 2). CIBS3 was obtained from late Holocene (>1000 years) beach sand from North Beach and CIDS1 was collected ~20 cm below the surface of a modern dune at West Beach (Fig. 1). Twenty-four aliquots of each sample were exposed to direct sunlight for two days and then given known laboratory doses (~6 Gy for CIBS3 and ~1 Gy for CIDS1). Weighted mean measured-to-given dose ratios of 1.00 \pm 0.20 and 1.11 \pm 0.01 were obtained for CIBS3 and CIDS1, respectively. Several

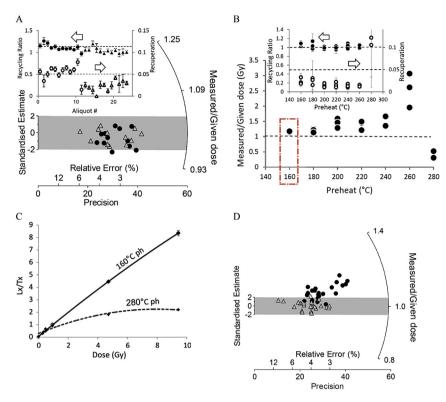


Fig. 2. A) Dose recovery tests for sample CIDS1 conducted with (triangles), and without (circles) a 40 s hotwash at 180 °C. Note that recuperation (inset graph) decreases when a hotwash is applied. B) Preheat plateau test results for sample CIDS1. Recycling ratios and recuperation rates for the preheat plateau test are in the inset graph. C) Dose response curves generated using a preheat of 160 °C and a preheat of 280 °C. A high preheat leads to degradation of the signal and distortion of the dose response curve. D) Dose recovery test results for feldspar from sample (CIDS1) after bleaching with direct sunlight and administering a laboratory dose of ~1 Gy (solid circles). The same dose recovery test was repeated except the sample was bleached with IR light in the laboratory for 500 s (triangles).

aliquots from the younger sample yielded measured-to-given ratios greater than unity (Fig. 2D). Huntley and Clague (1996) observed that the IR-excited luminescence of K-feldspar extracted from recently deposited tsunami-lain sand at Tofino, Vancouver Island was significantly brighter (by up to $2\times$) for grains that were exposed to direct sunlight than it was for those that were exposed to filtered sunlight lacking high-energy wavelengths. They concluded that the high-energy portion of sunlight can cause phototransfer of charge to the traps being sampled during measurement either from other traps, or directly from the valence band. This may lead to over-estimates of the D_e , particularly in young samples.

Another dose recovery test was carried out on sample CIDS1 after bleaching with IR diodes (rather than direct sunlight) for 500 s (Fig. 2D). The measured-to-given dose ratio obtained 1.00 \pm 0.01, which suggests that a portion of the luminescence signal measured from natural K-feldspar in these samples may be a result of phototransferred charge. Lian and Huntley (1999) was unable to detect phototransfer in K-feldspar from the Fraser River valley, southwest BC, suggesting that this effect is not always present, and therefore should be evaluated on a site-by-site basis.

4.3. Anomalous fading

In luminescence dating studies, experiments to determine the severity of anomalous fading in feldspar are not always undertaken, or if they are, they are often measured from a small subset $(\sim 1-3)$ of samples (e.g., Lian et al., 1995; Wolfe et al., 2008). In this study we corrected each individual sample using its own fading rate (g-value, as defined by Huntley and Lamothe, 2001) so that sample-to-sample variability in fading rates can be assessed and accounted for.

Auclair et al. (2003) devised a method that allows the fading rates of individual feldspar aliquots of a sample to be measured over a time period of ~3-5 days. This method involves giving an aliquot a known laboratory dose, then preheating and measuring the sensitivity-corrected signal (L_X/T_X) of the same aliquot several times using a range of delay periods between irradiation and L_x/T_x measurement. This method is expected to avoid scatter in fadingcorrected age (or D_e) distributions that may be due to aliquot-toaliquot variability in fading rates. It should also allow for more accurate age estimates from fading-corrected aliquot age distributions using appropriate statistical models such as the Minimum Age Model (MAM) for sediments that have not been sufficiently exposed to sunlight before burial (Galbraith et al., 1999), the Central Age Model (CAM) for well-bleached deposits (Galbraith et al., 1999), or the Finite Mixture Model (FMM) (Roberts et al., 2000) for sediments containing a mixture of old and young grains from different sedimentary units (e.g., Neudorf et al., 2012, 2014a, b).

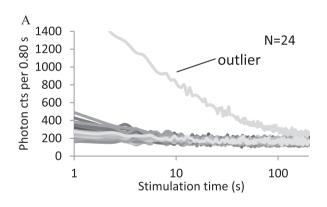
Given the high number of samples of interest in this study (17), an experiment was conducted to see if measurement time could be reduced by correcting the age obtained from the CAM (or MAM) weighted mean D_e value of 24 aliquots of a sample using a weighted mean g-value from the measured fading rates of a subset of only 12 aliquots. To this end, two methods of age determination were applied to sample CIBS3, and the results are compared in Table S6. Method #1 involves correcting the ages obtained from CAM and MAM weighted mean D_e values using the weighted mean g-value from 12 aliquots. Method #2, the more rigorous and time-consuming method, involves correcting the individual aliquot ages using their individual fading rates, then calculating the MAM and CAM weighted mean fading-corrected D_e and dividing this value by the environmental dose rate. A typical fading plot for one

aliquot of CIBS3 is shown in Fig. S2 and the g-value distribution of the same sample is shown in the inset graph. All fading corrections were conducted using the model of Huntley and Lamothe (2001). CAM age estimates obtained using methods 1 and 2 are consistent with each other within one standard deviation. MAM age estimates using methods 1 and 2 are consistent with each other within two standard deviations (Table S6). Interestingly, correcting the age of each individual aliquot using its own fading rate did not decrease the overdispersion in this sample (see footnote of Table S6). 'Overdispersion' (OD) is the spread in D_e values remaining after all measurement uncertainties have been taken into account (Galbraith et al., 2005; Galbraith and Roberts, 2012). Other potential sources of OD for this, and all other samples are listed in Section 4.5. Therefore, based on this result, and to save measurement time, age estimates derived from the weighted mean (CAM and MAM) D_e values of all Calvert Island samples were corrected for fading using the weighted mean g-value of 12 aliquots for each sample.

Weighted mean fading rates of all 17 Calvert Island samples are plotted in the histogram in Fig. S2 and listed in Table S7 (column 7). Fading rates vary from sample to sample and range from 4.70 ± 0.15 to $8.75\pm0.16\%$ per decade. Fading rates of K-feldspar from dune sands on Haida Gwaii and tsunami-lain sands and intertidal sediments from Vancouver Island fall into a slightly tighter, but comparable range of 4.9 ± 0.9 to $5.5\pm0.5\%$ per decade (Huntley and Clague, 1996; Huntley and Lamothe, 2001; Wolfe et al., 2008).

4.4. Correcting for phototransfer

The amount of phototransfer that occurs in K-feldspar grains in subaqueous environments, where UV rays are largely absorbed by



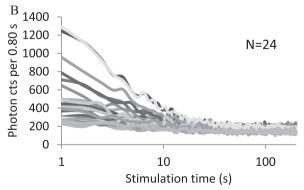


Fig. 3. A) IRSL signals from K-feldspar grains collected under ~40 cm of water in the nearshore zone. B) K-feldspar grains exposed to daylight under overcast skies for ~3 h. 'N' is the number of aliquots measured. Stimulation time is shown using a log time scale in all graphs. The D_e has been measured from all aliquots using SAR protocol 2 in Table 54, and CAM weighted mean estimates are 0.09 \pm 0.01 Gy (excluding the outlier) and 0.16 \pm 0.02 Gy for (A), and (B), respectively.

the water column, is thought to be less than what occurs in subaerial environments (Huntley and Clague, 1996; Ollerhead and Huntley, 2011). To further test this hypothesis on Calvert Island K-feldspar, a sample (CIMB 7) was collected from the ground surface in the nearshore zone of West Beach under 40 cm of water. The natural IRSL signal and D_e was measured from 24 aliquots of CIMB 7, as well as an additional 24 aliquots of this sample that was exposed to ~3 h of direct daylight (Fig. 3). As expected, the IRSL signals of the daylight-exposed grains are significantly brighter than those of the non-exposed grains, supporting the hypothesis that UV light exposure leads to an increase in signal intensity in these samples (cf. Huntley and Clague, 1996).

If we assume that the difference (0.07 \pm 0.02 Gy) between the weighted mean CAM D_e value of the daylight-exposed grains and the non-exposed grains approximates the difference in D_e that would result from phototransfer, this value can be subtracted from D_e estimates from dune sands that presumably would have been most susceptible to UV light exposure. This correction was tested on two modern samples: one collected ~20 cm below the surface of a modern dune at West Beach (CIDS1) and one collected from a foredune remnant on the east side of Wolf Beach (CIDS11). Before a correction for phototransfer is made, CIDS1 gives a fadingcorrected (CAM) age of 54 ± 6 years and CIDS11 gives a fadingcorrected (CAM) age of 56 ± 5 years. When we subtract the phototransferred dose from the weighted mean (CAM) D_e values, we get "phototransfer-corrected" age estimates of 4 ± 14 years, a value much closer to that expected. When the phototransfer correction is applied to the MAM D_e value of sample CIDS1 (see the rationale for the use of the MAM for this sample in Section 4.5, below), a phototransfer-corrected D_e value of -0.02 ± 0.02 Gy is obtained. This value is consistent with zero within one standard deviation. Using this method, phototransfer-corrected age estimates have been calculated for all dune samples and samples CIDBS5 and 6, which may also consist of aeolian sediment.

4.5. Refined age estimates and consistency with radiocarbon chronology

Fading-corrected ages have been calculated for all optical dating samples using the refined SAR protocol (protocol 2) and all age estimates and OD values are listed in Table S7. Recuperation values are generally satisfactory (less than 10%) for all but four samples from which ages were obtained (CIBS2, CIBS6, CIDS1 and CIDS11, Table S7). When all aliquots with high (>10%) recuperation are rejected in these samples, their age estimates increase by ~10-~40 years, but these ages should be treated with caution, given the low number of accepted aliquots. Phototransfer-corrected age estimates for (subaerial) dune samples are shown in parentheses in Table S7. Sources of OD may include differences in β dose received by individual grains in their burial environment due to proximity to pore water, cemented grain clusters, or organic matter (e.g., Lian et al., 1995; Murray and Roberts, 1997; Lian and Huntley, 1999), insufficient or heterogeneous exposure of some grains to sunlight before burial (e.g., Olley et al., 1996, 1997), and/or post-depositional intrusion/mixing of younger grains into older deposits or vice versa (e.g., Roberts et al., 1998, 1999; Jacobs et al., 2008). Because our samples were obtained from relatively massive sand units (sometimes overlain by organic-rich units), and because our external dose rates only constitute ~50-70% of the total dose rate of our samples due to the high internal dose rate contribution of ⁴⁰K inside K-feldspar grains, it is expected that heterogeneities in the external β dose contribution are minimal in most cases.

As expected, OD values of the beach samples (typically between 25 and 50%) tend to be higher than those of the dune samples suggesting that some waterlain K-feldspar grains did not have

sufficient sun exposure prior to burial to completely deplete their signal. Research elsewhere has shown that OD values for single-grain and single-aliquot datasets of quartz samples known or thought to have been fully bleached at burial and not affected by post-depositional disturbance (or by significant differences in β dose rate among grains buried at the same time) commonly have OD values of ~10–20%, with some reported examples exceeding 30% (Galbraith et al., 2005; Jacobs and Roberts, 2007; Arnold and Roberts, 2009). Here, we assume that this result would apply to K-feldspar as well. All but one (CIDS1) of the Calvert Island dune samples have OD values less than 25% suggesting that few K-feldspar grains in these samples suffered from incomplete bleaching before burial. Weighted mean D_e values were calculated using both the CAM and MAM models for all samples with OD values greater than 25% (Table S7).

Our refined age estimates show consistency with radiocarbon age control and illustrate how optical age estimates can help identify sites where radiocarbon-dated organic-rich units have been reworked. Phototransfer-corrected CAM ages of 3503 \pm 318 and 3710 \pm 304 years from Cobble Beach sands (CIDBS 5 and 6) are stratigraphically consistent with an AMS radiocarbon age of 7580-5301 years cal BP from an underlying peat bed (Fig. S3A, Table S2). A CAM age of 2885 ± 287 years from CIBS3 (North Beach) is consistent with AMS radiocarbon ages of 5893-5802 years cal BP from a cone and a fragment of bark from the base of an underlying peat bed, and 1862–1464 years cal BP from shell from the base of an overlying shell midden (Fig. S3B, Table S2). The MAM age $(1173 \pm 130 \text{ years})$ of this sample, however slightly underestimates the radiocarbon age of the base of the overlying midden, and the reason for this is unclear. If the radiocarbon age is accurate, it is possible that the MAM model takes into account grains that have received lower than average radiation doses as a result of a heterogeneous radiation field induced by adjacent organic matter, calcium carbonate and/or pore water. Because we only used a subsample of the bulk sediment for dating for measurements of the sample dose rate, this may not have been accounted for by our dosimetry measurements.

Sample CIBS4 was collected at North Beach from ancient beach sediment which is capped by a ~10 cm-thick organic-rich bed (peat and sand); the peat and sand bed is directly overlain by about 1 m of modern beach sediment (Fig. S3C). Sand lenses within the peat unit, as well as macrofossils extracted from the peat bed suggests that it consists of detrital material that was likely reworked from a peat unit exposed in the beach scarp ~20 m away, which yielded a radiocarbon age of 5893–5802 years cal BP. A macrofossil collected from the peat bed at the location of CIBS4 returned a radiocarbon age of 6740–6654 cal yrs BP, and CIBS4 returned optical ages of 5578 \pm 515 years (CAM) and 3527 \pm 372 years (MAM), which are younger than the overlying peat (Fig. S3C, Table S2). This site demonstrates the utility of optical dating in helping to identify sites where radiocarbon-dated organic matter is not *in situ* leading to an age over-estimate.

5. Conclusions

This study describes the luminescence characteristics of Calvert Island quartz and K-feldspar and, for the first time, a SAR procedure is developed and tested for K-feldspar on the central coast of BC. K-feldspar samples from Calvert Island are best suited to SAR protocols using low preheat temperatures and a hotwash treatment. Bleaching tests show that the high-energy portion of the sunlight spectrum causes phototransfer of charge from non-light sensitive traps to traps sampled during $D_{\mathcal{C}}$ measurement and we have devised and implemented a method of measurement and correction for phototransfer based on experiments on modern

subaqueous and subaerial samples. Anomalous fading tests show appreciable sample-to-sample variability in anomalous fading rates implying that, in this region, it is not sufficient to rely on two or three representative fading rates as has been done in some studies elsewhere.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.quageo.2014.12.004.

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