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Carolina Barbosa Leite da Cruz Luminescence signals sensitivity of quartz from the Putumayo foreland basin, north-eastern Andes (Colombia): implications for sediment provenance and basin evolution

October 2023

Institute of Geosciences, University of São Paulo, São Paulo, Brazil

Degree: Master Supervisor: Prof. Dr. Fabiano do Nascimento Pupim

Sediment provenance analysis is an approach widely used to reconstruct the set of source rocks of a rock/sedimentary deposit, which allows the study of source-to-sink processes, sedimentary basin filling, and depositional environments. Moreover, it helps to understand tectonic systems, magmatic processes, and climatic conditions in the source areas. The Putumayo Sedimentary Basin in the northeast of the Andes (Colombia) is a key area for hydrocarbon exploration and provides a comprehensive and uninterrupted sedimentary record from the Cretaceous to the Cenozoic. Despite its considerable geological and economic importance, there is a lack of knowledge about the origin of the sediments that fill this basin. The scope of this work was to test the feasibility of using optically stimulated luminescence (OSL) signals to distinguish sedimentary units and to track sedimentary provenance in the Putumayo Foreland Basin. The method consisted in (i) calculating the relative sensitivity of quartz OSL signals (%BOSL<sub>F</sub>), which is given by the ratio of net signal within the first second of the quartz OSL decay curve (presumably dominated by the fast component) to the net signal of the total OSL decay curve; and (ii) assessing the ratio between the intensity of feldspar signals and quartz signals (IRSL<sub>[1.2s]</sub>/BOSL<sub>[1s]</sub>). In general, all lithostratigraphic units showed low %BOSLF and high IRSL<sub>[1.2s]</sub>/BOSL<sub>[1s]</sub>, indicating source areas with rocks with a low number of sedimentary cycles and high denudation rates, as expected for the Andean orogen. However, slight differences between lithostratigraphic units could be identified. The sediments of the Cretaceous rocks (Caballos and Villeta Fms) showed very low %BOSL<sub>F</sub> and relatively high IRSL<sub>[1.2s]</sub>/BOSL<sub>[1s]</sub>, indicating that their source areas are composed of metamorphic and igneous rocks, characterized by the first sensitization cycle and higher feldspar content, related to the Amazon Craton (east) and the Paleo-Central Cordillera (west). An abrupt increase in %BOSL<sub>F</sub> and decrease in IRSL<sub>[1.2s]</sub>/BOSL<sub>[1s]</sub> occurs across the K-Pg boundary (i.e., a thin layer of sediment deposited during the Cretaceous-Paleogene extinction event), suggesting that the Cenozoic rocks (Rumiyaco, Neme and Pepino Fms, and Orito Gr) received sediments reworked from Mesozoic and Proterozoic rocks due to the tectonic uplift of the Eastern Cordillera and the unroofing process during the Cenozoic. In conclusion, this work shows that luminescence sensitivity has high potential as a sediment provenance tool for interpreting basin evolution, even in low-contrast source areas.

A PDF of this thesis can be downloaded from: https://www.teses.usp.br/teses/disponiveis/ 44/44142/tde-12122023-081054/pt-br.php

A part of this work was presented at the 17<sup>th</sup> International Luminescence and Electron Spin Resonance Dating Conference (25–30 June 2023).

# Scott K. Fitzgerald Investigating stimulated luminescence within complex silicates to explore charge mobility and trap distribution January 2024

SUERC, University of Glasgow, Glasgow, United Kingdom

Degree: MSc by Research

#### Supervisor: Prof. Dr. David C.W. Sanderson, Dr. Alan J. Cresswell, Dr. Loïc Martin

The mechanisms behind charge storage and transport within feldspars are of key research interest to explain and model the observed behaviours of irradiated feldspars following stimulation. There is no general consensus on whether infra-red stimulated luminescence (IRSL) in feldspars is due to a single fundamental trap or multiple traps, or whether relaxation mechanisms are tunnelling derived or charge movement through band-tail states and conduction band.

Four separate investigations, using 22 feldspar samples which were previously used in several studies of feldspar luminescence, from four feldspar families (K-Feldspar, Albite, Plagioclase and Microcline samples as well as the F1 IAEA standard material), have been conducted to examine how post-IRSL phenomena can be used to model charge storage and transportation within the material lattice of feldspars. The investigations included the measurement of both post-IR poststimulation phosphorescence (PSP) and post-IR phototransferred thermoluminescence (PTTL) at different temperatures and following different stimulation wavelengths to characterise variability in relaxation times on the order of microseconds to minutes.

This work highlights the intrinsic complexities of the lattices of feldspars, noting that most changes made appeared to have a measurable effect on either the decay lifetimes or the PTTL peak activation energies or lifetimes. This implies that a distribution of trapping systems must exist within the lattice to allow the observed variability in behaviour. The decay lifetimes of the post-IR PSP decay corresponded to the lifetimes of the PTTL peaks, suggesting a relationship between the two phenomena. This was then exploited by first modelling the potential activation energies of PTTL peak lifetimes that were approximately equal to the computed PSP decay component lifetimes at room temperature. A cryogenic investigation then confirmed that these predicted PTTL peaks were observed, for the first time, in feldspars.

These results demonstrate that, within feldspars, post-IR PTTL and PSP are representative of charge storage in multiple trap systems and transport of charge can be described by band-tail state movement post-stimulation, with no evidence of significant tunnelling.

A PDF of this thesis can be downloaded from: https: //theses.gla.ac.uk/84101/

# Monika Devi Multispectral Luminescence Studies: Methodological Developments and Applications April 2024

Physical Research Laboratory, Ahmedabad, India

Degree: Ph.D. Supervisor: Dr. Naveen Chauhan

In luminescence dating, quartz and feldspar are the most commonly used minerals. Over the past decade, luminescence dating using feldspar has been studied extensively due to its higher luminescence sensitivity and saturation dose ( $\sim$ 1200 Gy), which is approximately five times higher compared to quartz. This offers the potential to increase the range of luminescence dating up to millions of years. Its use has, however, been limited due to anomalous fading of the luminescence signal, which is athermal loss of signal in addition to that predicted by the thermal kinetic considerations. The luminescence production mechanism of feldspar is complex due to its complex defect structure and is not yet fully understood.

This thesis rigorously investigated aspects of the luminescence mechanism in feldspar and studied the traps and recombination centres responsible for luminescence. We explored the various spectral regions of feldspar, assessing their athermal stability through different stimulation and emission wavelength bands. Among all signals, the post violet IR stimulated luminescence (pVIRSL) signal resulted in the lowest average fading value of  $0.0 \pm 0.1$ % per decade. This indicates that the pVIRSL signal originates from stable electron and recombination centres. The TL and OSL experiments suggest that pVIRSL is a recuperated signal arising from the eviction of charges from deep traps by violet stimulation and their recapture by the IR trap. The study showed the role of deep traps present beneath the principal trap in pVIRSL emission.

Further, the pVIRSL signal was characterised for its dosimetric properties. A single aliquot regenerative dose protocol for the pVIRSL (pVIR-SAR) was developed for the estimation of equivalent doses. The proposed protocol fulfils all the essential criteria for the use of a SAR protocol, such as rapid bleaching (<10% of the equivalent dose in 60 min of daylight exposure), recuperation <5%, reproducibility within 10%, and dose recovery within 10%. The pVIR-SAR protocol was tested for several K-feldspar samples from varied depositional environments with age ranges between 6 to 286 ka. The obtained ages were consistent with the expected ages.

Further, the pVIR-SAR protocol enabled the dating of polymineral fine- and coarse-grain samples, as high energy violet stimulation bleaches the signal from quartz and permits probing of the pVIRSL signal only from feldspar. Therefore, the protocol allows the dating of unseparated polymineral samples and quartz separates containing feldspar as inclusions. The ages obtained from polymineral fine- and coarse-grain samples, which include volcanic ash, pottery, and fluvial samples, were consistent with geological reasoning and available age controls. The work suggests that mineral separation is not required if the pVIR-SAR protocol is used for dating.

Additionally, this thesis examined the application of pVIRSL and IRSL for luminescence dating of old exposed rock surfaces (>1 ka). Theoretical modelling has suggested that the rock surface exposure dating technique could potentially be applied to rocks up to 1 Ma old. However, in practice, the technique has yielded reliable ages primarily for younger samples (<1 ka). For older rock samples, underestimation in the ages is reported. The same is observed in the present thesis work for old glacially polished samples. Therefore, possible reasons for such a large discordance were explored. Scanning electron microscope with energy dispersive spectroscopy (SEM-EDS) analysis showed the presence of desert varnish and micro-fractures in the rock samples. The sample used for calibration of the model, however, had no desert varnish or micro-fractures, indicating that the parameters derived from the calibration sample were not suitable for estimating the ages of the unknown age samples. It also highlights prospects for refining luminescence dating techniques and addressing the complexities associated with surface exposure dating of polished rock samples. In conclusion, the thesis work contributes to the advancement of luminescence dating by expanding our understanding of feldspar luminescence and offering a viable alternative for dating various geological samples. It also sheds light on the challenges and complexities associated with dating exposed rock surfaces, highlighting the need for continued refinement of luminescence dating techniques in these contexts.

A PDF of this thesis can be obtained by contacting the author: monikaparmar678@gmail.com.

### Priyanka Singh High resolution Late Quaternary climate reconstruction from Higher Himalaya: A multi-proxy approach

May 2024

Birbal Sahni Institute of Palaeosciences, Lucknow, India

Degree: Ph.D. Supervisor: P. Morthekai (BSIP, Lucknow), Prof. Kuldeep Prakash (BHU, Varanasi)

This thesis emphasizes on two aspects, namely 1) chronology, and 2) palaeoclimate in the Higher Himalaya particularly focussed on the delineation of the contribution of the Western Disturbance (WD) from that of the Indian Summer Monsoon (ISM).

A 4 m proglacial palaeolake profile in Kunti Banar Valley near Kalla Glacier in the Higher Himalaya was taken for a high resolution (at 1 cm) palaeoclimate reconstruction. Both luminescence dating (fine grain polymineral) and <sup>14</sup>C AMS dating (bulk sediment organic carbon calibrated using Int-Cal20) methods were employed. An attempt was also made to date micro-feldspar inclusions within quartz grains by emphasizing more on the dose rate calculation. Apparently erroneous sets of ages were produced on two grounds. First, both radiocarbon and luminescence ages of the intermediate depth samples (180–235 cm) were conspicuously older than downward samples. Second, there was a systematic overestimation of radiocarbon ages compared to the luminescence counterpart.

The systematic overestimation of radiocarbon ages was explained by the presence of petrogenic organic carbon  $(OC_{petro} = 0.06\%)$  in the sediments. Raman spectrometry confirmed that the petrogenic organic carbon is present in the form of graphite. Overestimation of intermediate luminescence ages can be understood by the enhanced ISM during 10-6 ka, supported by various proxies, that brought sediments rapidly to the lake and caused the overestimation of luminescence ages as the sediments were poorly bleached. During the last  $\sim 16$  ka, the studied lake has buried 2.5 Gg  $OC_{petro}$  at an average burial flux of 160 (± 40) kg  $OC_{petro}$  a<sup>-1</sup>. This study suggests not to rely on bulk <sup>14</sup>C ages in Himalayan regions where the total organic carbon (TOC) is low. The palaeoclimate for the past  $\sim 16 \text{ ka} (15.3 \pm 3.0 \text{ ka})$ was reconstructed using the stable carbon isotopic composition ( $\delta^{13}$ C), TOC in the sediment, environmental magnetism variables, end member modelled grain size data, palynofacies, n-alkane data and geochemical proxies. The intermediate samples were of the period (11.6-4.9 ka) where the post Younger Dryas (YD) intensification of the ISM and

the Holocene Climate Optimum (HCO) occurred. The linear ordination analysis suggested that the variability among the profile samples was primarily controlled by one component (45.5%) and that could be identified as precipitation (PCA 1). Considering the  $\delta^{18}$ O from the Dongge Cave (China) and sea surface temperatures (from the Bay of Bengal) as explanatory/predictor variables, redundancy analysis suggested that the precipitation (91.8%) and the temperature (8.2%) could be significantly identified as or associated with the first and second PCA axes. The PCA 1 scores were found significantly causally (Granger Cause) related to  $\delta^{18}$ O of the Dongge Cave. These results suggested that the climate variability as recorded in the studied lake sediment was primarily controlled by hydrological conditions (rain water, snow melt water or/and glacial melt water) in the catchment area. Long term palaeoclimate reconstruction for  $\sim 80 \text{ ka}$ was done using moraines of the Kunti Banar valley. There were three stages of moraine formation in the time periods of 17-16 ka, 26-21 ka, and 81-41 ka. Thus a non-continuous long-term palaeoclimate was reconstructed during 80-16 ka using moraines.

The delineation of the WD and the ISM was done by applying non-linear time series such as recurrence quantification analysis (RQA), recurrent network analysis (RNA) and Laplacian eigenmaps for recurrence matrices (LERM). These methods were validated by applying them to artificially created data from a logistic map (where a periodic and chaotic time series were mixed) and two sets of Gaussian noise data. RQA and LERM methods suggest that the WD was dominant during 12.5-11.0 ka, and this time period is collinear with the timing of the global YD event. During 11–9 ka, the ISM was dominant following the YD. Then, during 9-7 ka, again the WD became dominant, but the model suggested that along with the WD, the ISM was also influencing during 8.0–4.2 ka. After  $\sim$ 4 ka towards the present, the WD has been dominating. There were three climatic transitions observed at 8.2 ka, 4.2 ka and 2.3 ka, with the 8.2 and 4.2 ka events being related to the well-known global dry and cold events. The time series analysis was able to delineate the two monsoon systems but it needs more robust time series and a better proxy for the WD.

A PDF of this thesis can be obtained by writing to priyankaasingh10may@gmail.com.