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Investigation of luminescence signals of quartz and their application for provenance analysis and extending dating range of Brazilian fluvial sediments

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Dedication

This thesis is dedicated to my wife

Belyse Ingabire Niyonzima,

My Daughters

Belinda Shami Niyonzima,

Abby Liz Hayla Niyonzima

Gabby Julia Kayla Niyonzima

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Abstract

This work investigates the feasibility of applying quartz radiofluorescence (RF) as provenance proxy, carried out experiments to evaluate if violet stimulated luminescence (VSL) of quartz is able to extend the dating age range of fluvial sediments in Brazil, and performed optically stimulated luminescence (OSL) dating to establish formation ages of ferruginous deposits (ironstones) of the Xingu River in Eastern Amazonia. The focus on luminescence properties of quartz from fluvial deposits is motivated by their widespread occurrence across Brazil, land of large rivers, thus, representing the most important continental record of landscape changes during the Quaternary.

Firstly, the work examines RF emissions in quartz from parent rocks (igneous plutonic and volcanic) and sediments of different provenances. After deconvolution of RF spectra into its emission bands, emission band intensities were plotted versus OSL (first 1s) and 110°C thermoluminescence (TL) peak sensitivities to investigate whether RF emission band intensity can be used as sediment provenance. It has been shown that ultraviolet (UV) RF intensity correlates with both OSL and TL sensitivities of quartz from sediments. This correlation supported the suggestion that both 110°C TL peak and OSL of quartz use the same recombination centers rather than the same electron trap and that sensitization processes in nature of two signals might be due to the changes in recombination probability instead of changes in charge trapping probability. It is concluded that UV-RF intensity measured using X-ray sources can also be used for provenance analysis of sediments in the same way as the OSL and TL sensitivities.

Characteristics of VSL signals of quartz from major fluvial systems from Western Brazil (Pantanal), Southeastern Brazil (Paraná River basin) and Central and Eastern Amazonia were investigated to assess if the VSL signal is suitable to extend the age range of luminescence dating of fluvial sediments. Single-aliquot regenerative dose (SAR), multiple aliquots regenerative dose (MAR) and multiple aliquots additive dose (MAAD) protocols were used to estimate the maximum dose that can be estimated by VSL signals for Brazilian sediments. Quartz VSL was found to significantly increase the dating limit to the Early Pleistocene (~800 ka to 1.6 Ma for the $2D_0$ MAAD) even with the relatively lower characteristic doses (D_0) observed in dose response curves of the studied samples. Despite the improvement in luminescence age range of Brazilian fluvial sediments using VSL dating

relative to the conventional OSL dating, the behavior of VSL appears to be sample dependent, with natural VSL signal missing in samples from Amazonian settings.

In the estimation of formation ages of ironstones from the Xingu River, ages between ~60 ka and 3 ka were obtained using the SAR OSL dating protocol. This indicates that ironstones of the Xingu River result from a Late Pleistocene and Holocene surface geochemical system able to precipitate goethite and cement fluvial sediments under transport. The high dose rates (2.7-12.3 Gy ka⁻¹) of these fluvial deposits limits the OSL dating to the last ~60 ka, assuming maximum doses of ~200 Gy estimated using quartz OSL applied to fluvial deposits across Brazil.

The performed investigations improve the applicability of luminescence signals (RF, OSL and VSL) to obtain provenance and depositional age data from a variety of fluvial deposits occurring over Brazil. This is a contribution to expand the application of luminescence methods to study fluvial deposits.

Keywords: Radiofluorescence; Optically stimulated luminescence, Ironstones; Violet stimulated luminescence; Fluvial sediments.

Resumo

Este trabalho investigou a viabilidade da aplicação de radiofluorescência (RF) do quartzo como indicador (*proxy*) de proveniência, realizou experimentos para avaliar se a luminescência do quartzo estimulada por luz violeta (VSL, em inglês) é capaz de estender o intervalo temporal da datação de sedimentos fluviais no Brasil e realizou datações por luminescência opticamente estimulada (OSL, em inglês) para estabelecer idades de formação de depósitos ferruginosos (*ironstones*) do rio Xingu na Amazônia Oriental. Assim, esta tese de doutoramento buscou desenvolver aplicações das características de luminescência do quartzo no estudo de depósitos fluviais quaternários. Isto foi motivado pela ampla ocorrência de depósitos fluviais no Brasil, terra dos grandes rios, os quais geraram importante registro continental de mudanças de paisagem e do clima durante o Quaternário.

Inicialmente, o trabalho examinou as emissões de RF em quartzo de rochas fonte (ígneas plutônicas e vulcânicas) e sedimentos de diferentes proveniências. Após a deconvolução dos espectros de RF em suas bandas de emissão, as intensidades das bandas de emissão foram comparadas com as sensibilidades OSL (primeiro 1s) e termoluminescência (TL, pico a 110°C) para investigar se a intensidade da banda de emissão de RF pode ser usada como indicador de proveniência sedimentar. Foi demonstrado que a intensidade de RF ultravioleta (UV) se correlaciona com as sensibilidades OSL e TL do quartzo de sedimentos. Essa correlação apoiou a sugestão de que tanto o pico TL de 110°C quanto o OSL de quartzo usam os mesmos centros de recombinação em vez da mesma armadilha de elétrons e que a sensibilização natural dos sinais OSL e TL pode ocorrer devido às mudanças na probabilidade de recombinação em vez de mudanças na probabilidade de captura de cargas. Conclui-se que a intensidade de UV-RF medida com fonte de raios X também pode ser utilizada para análise de proveniência de sedimentos da mesma forma que as sensibilidades OSL e TL.

Características dos sinais VSL de quartzo dos principais sistemas fluviais do Oeste do Brasil (Pantanal), Sudeste do Brasil (bacia do Rio Paraná) e Amazônia Central e Oriental foram investigadas para avaliar se o sinal VSL é adequado para estender o intervalo de tempo da datação por luminescência de sedimentos fluviais brasileiros. Os protocolos de dose regenerativa de alíquota única (SAR, em inglês), dose regenerativa de alíquotas múltiplas (MAR, em inglês) e dose aditiva de alíquotas múltiplas (MAAD, em inglês) foram usados para estimar a dose máxima que pode ser medida por sinais VSL do quartzo presente em sedimentos quaternários brasileiros. Conclui-se que o sinal VSL do quartzo pode aumentar

significativamente o limite de datação até o Pleistoceno Inicial (~800 ka a 1,6 Ma para $2D_0$ obtida por MAAD), mesmo com doses características relativamente mais baixas (D_0) observadas nas curvas de dose-resposta das amostras estudadas, se comparadas com outros estudos da literatura. Apesar da VSL ampliar o intervalo de tempo da datação por luminescência dos sedimentos fluviais brasileiros, em relação à datação OSL convencional, o comportamento do sinal VSL parece ser dependente da origem amostra. Isto é sustentado pela ausência de sinal VSL natural em amostras de quartzo derivadas dos depósitos fluviais amazônicos estudados.

Na estimativa das idades de formação dos *ironstones* do rio Xingu, idades entre ~60 ka e 3 ka foram obtidas por meio do protocolo de datação SAR OSL em alíquotas de quartzo. Isso indica que as rochas ferruginosas do rio Xingu resultam de sistema geoquímico superficial do Pleistoceno Tardio e Holoceno, capaz de precipitar goethita e aprisionar sedimentos detríticos em transporte no canal do rio Xingu. As altas taxas de dose (2,7-12,3 Gy ka⁻¹) desses depósitos fluviais limitam a datação OSL para os últimos ~60 ka, assumindo doses máximas de ~200 Gy estimadas a partir da dose característica (D_0) das curvas de dose resposta.

As investigações realizadas contribuem para entender a sensibilização natural de sinais OSL e TL, processo ainda pouco compreendido, e ampliam as aplicações dos sinais de luminescência (RF, OSL e VSL) do quartzo para análise de proveniência e geocronologia de depósitos fluviais no Brasil.

Palavras-chave: Radiofluorescência; Luminescência opticamente estimulada, Rochas sedimentares ferruginosas; Luminescência estimulada por luz violeta; Sedimentos fluviais.

Introduction

Motivation and purpose

Human excellence is nurtured by inherent curiosity to know about the past (Gould, 1987). The age determination of geological and archeological events in the last ~2.6 million years (The Quaternary period) is critical for understanding past climate and environmental changes, landscape evolution, and human evolution and dispersal. This understanding is important for future predictions of climate, environmental changes and resource planning.

In this connection, the luminescence dating technique, a novel and rapidly developing application in geochronology, has played an important role in age determination of the archaeological and geological events in the Quaternary timespan (Aitken, 1998; Rhodes, 2011; Liritzis *et al.*, 2013; Roberts *et al.*, 2015; Duller, 1997). In particular, it has proven to be a highly useful and widely applicable technique for obtaining chronologies for the Late and Middle Pleistocene (Buylaert *et al.*, 2008; Rittenour, 2008; Cunha *et al.*, 2012; Herman *et al.*, 2013; Guralnik *et al.*, 2015; Henshilwood *et al.*, 2002; Singhvi and Porat, 2008; Fattahi and Walker, 2007; Wallinga, 2002).

Luminescence dating is an interdisciplinary research field based on solid state physics, radiation physics, and Earth and archaeological sciences; all these areas have contributed to the development of the luminescence dating technique. Despite the immense success of the luminescence technique, there are continuous attempts from laboratories across the world to further improve the technique for wider application. Some of the active research areas are: use of luminescence as a sediment tracer and provenance tool (Sawakuchi *et al.*, 2018; Gray *et al.*, 2019), extending the age range to cover the full Quaternary (Ankjaergaard *et al.*, 2013), investigating the feasibility of luminescence method to shed the light on the formation age of Quaternary rocks (Niyonzima *et al.*, 2022), and modelling charge build up in thermally and optically dynamic environments (King *et al.*, 2016). Even a slight improvement in the method can have a significant positive impact on the application of the technique in the Geosciences or Archaeology fields, and on the age resolution of the past events.

The researchers rely on the fact that natural minerals such as quartz and feldspar, which are widely used in dating, are rich in luminescent defects and have complex charge transfer processes. It is expected that a better understanding of these defects and processes will give

rise to more robust dating methods, and help to place the existing dating protocols on a solid foundation.

Hence, the first overriding objective of this thesis, therefore, was to investigate luminescence signals of quartz extracted from Brazilian fluvial sediments for better understanding of charge transfer process, and the application of the technique for age resolution and improving of its dating range. This consisted of investigating the variation of the radiofluorescence (RF) emission spectra in quartz extracted from different igneous rocks and sediments of different geological settings. We investigated the difference in RF emission spectra between quartz extracted from igneous rocks and sediments for better understanding the mechanisms involved in thermoluminescence (TL) and optically stimulated luminescence (OSL) sensitivities, properties successfully used in sediment provenance analysis (e.g. Sawakuchi *et al.*, 2018; Mendes *et al.*, 2019). The possibility of use of RF of quartz for sediment provenance analysis was also investigated by studying the correlation between the ultraviolet (UV) RF emission intensity and sensitivity of both TL and OSL emissions.

Besides the widespread unconsolidated sandy fluvial deposits, some bedrock rivers in eastern Amazonia host sandy and gravelly deposits cemented by goethite. These deposits form rocks over the riverbed and represent unique riverine landscape features. This is the case of the Xingu River draining shield areas in eastern Amazonia. Additionally, it has been reported that the original oxygen isotopes ratio of iron minerals such as goethite and hematite is generally preserved, thereby providing information about weathering and climate conditions at the time of formation (Giral-Kacmarcik *et al.*, 1998; Girard *et al.*, 2002). The formation ages of ironstones (fluvial sands and gravels cemented by goethite) of the Xingu River are unknown and their formation process is still poorly understood despite their relevance as substrate for the aquatic ecosystem (Fitzgerald *et al.*, 2018). Different dating methods of ironstones were applied in Brazilian settings, including (U-Th)/He of goethite (Riffel *et al.*, 2016), electron paramagnetic resonance of matrix kaolinite (Allard *et al.*, 2018) and cosmogenic nuclides of trapped quartz grains (^{10}Be) (Pupim *et al.*, 2015). These methods are suitable to obtain ages in the hundred thousand to million years timescale and dating of ironstones is still challenging for the thousand years timescale necessary to cover the late Pleistocene and Holocene age range. In this thesis, we checked the feasibility of OSL dating on detrital quartz grains within ironstones to shed light on the timing and processes of formation over the riverbed of the Xingu River. This was the second objective of this thesis.

The third objective aimed to extend the age range of luminescence dating to tackle research problems beyond the late Pleistocene, which is usually the age limit for quartz OSL dating. In this way, deposition ages of fluvial sediments have a fundamental role to constrain the landscape changes in Amazonia during the middle and early Pleistocene, when most species representing the modern Amazonian biota appeared (Smith *et al.*, 2014). Geochronological methods to determine reliable burial ages for the middle and early Pleistocene fluvial sedimentary record of Amazonia are crucial to understand the role of the physical landscape for the biota diversification (Hoorn *et al.*, 2017). So far, most successful dating studies of Amazonian Quaternary sedimentary deposits are based on radiocarbon dating of rare organic material (e.g. Rossetti *et al.*, 2005) or quartz optically stimulated luminescence (OSL) (e.g. Rossetti *et al.*, 2015). However, the use of quartz for OSL dating is typically limited to the last 100–150 ka due to saturation of the OSL signal around 150–200 Gy (Wintle and Adamiec, 2017). The violet stimulated luminescence (VSL) from quartz, which probes traps deeper than those accessible by blue light, has been observed to grow with doses up to about 1000 Gy (Jain, 2009), ~10 times higher than the OSL from the same grains. Further studies confirmed that VSL signal originates from thermally stable with lifetime of 10^{11} years at 10°C (Ankjaergaard *et al.*, 2013, 2015) with no athermal loss (Ankjaergaard *et al.*, 2013); thus giving the potential to extend the age range of quartz luminescence dating to cover the full quaternary period. Therefore, we explored the VSL single aliquot regenerative dose (SAR), multiple aliquots regenerative (MAR), multiple aliquots additive dose (MAAD) protocols, and investigated the behavior of the VSL signal under different measurement conditions with the aim of extending the dating range of luminescence dating for Brazilian fluvial sediments.

Thesis outline

The thesis is outlined in five chapters. Chapter 1 introduces the basic concepts of dating based on luminescence methods. The physical basics of luminescence, principal of luminescence, environments suitable for luminescence dating, and explanation of how the phenomenon can be used for dating is presented.

Chapters 2, 3 and 4 describe three manuscripts, where two are published (Niyonzima *et al.*, 2020, 2022) and other is in preparation for submission in peer-reviewed scientific journal. Each article is individually understandable. They are structured so that they describe the motivation, experimental procedures, results and discussion. They all address the subject of using luminescence signals from quartz extracted from Brazilian fluvial sediments for provenance analysis and dating.

The work presented in Chapter 2 is based on the article “*Radiofluorescence of quartz from rocks and sediments and its correlation with thermoluminescence and optically stimulated luminescence sensitivities*”, published in *Ancient TL* (Niyonzima *et al.*, 2020). This study investigates radiofluorescence (RF) emissions in quartz from parent rocks and sediments of different provenances and different optically stimulated luminescence (OSL) and thermoluminescence (TL) sensitivities. The correlation of OSL and TL (110°C peak) sensitivities were compared to RF emission peak sensitivities in order to understand the sensitivity variations in terms of charge traps and recombination centers. We investigate the relationships between intensities of RF emission peaks and OSL and TL sensitivities in order to investigate the possibility of RF emission band sensitivity to be used as a provenance proxy as in the way as OSL and TL sensitivities for quartz from sediments.

The work presented in Chapter 3 is based on manuscript: “*Testing the potential of quartz violet stimulated luminescence (VSL) for dating of Brazilian fluvial sediments*” (Niyonzima *et al.*, in preparation). On this chapter, VSL properties of quartz from major fluvial systems in South America, represented by fluvial terraces from western Brazil, southeastern Brazil and central and eastern Amazonia. Firstly, we investigated the presence of VSL signals in quartz from the studied samples under different preheating temperatures. Afterwards, dose recovery test of SAR-VSL protocol and thermal stability test were performed to evaluate dose estimation capacity. We also estimated equivalent dose (D_e) and characteristic doses (D_0) of samples that showed VSL signal using a single aliquot regenerative dose (SAR), multiple aliquots regenerative doses (MAR), and multiple aliquots

additive doses (MAAD) protocols. Considerations about the use of VSL to extend the age limit of quartz dating beyond the late Pleistocene were addressed in this chapter.

The work presented in Chapter 4 is based on the article “*Luminescence dating of quartz from ironstones of the Xingu River, Eastern Amazonia*”, published in *Quaternary Geochronology* (Niyonzima *et al.*, 2022). In this study, OSL dating method was applied for first time in establishing formation ages of ferruginous deposits (ironstones) of the Xingu River in Eastern Amazonia, Brazil. Equivalent doses, environmental dose rates and hence luminescence ages were determined. Equivalent dose distributions were also evaluated in order to inform about how quartz grains are incorporated into ironstones. Additionally, the organic content of some samples was dated by radiocarbon (^{14}C) for comparison with quartz OSL ages.

In Chapter 5, we summarize the main conclusions based on outcomes from the thesis. In this chapter also, we clarified the possible work to be done in the future in order to support some arguments raised in this work.

Summary conclusions

Chapters 2, 3 and 4 of this study provide characteristics of different luminescence signals (RF, VSL and OSL) of quartz from Brazilian fluvial rocks (ironstones) and sediments. All characteristics confirmed the use of the luminescence signals for provenance, first estimation of fluvial ironstones formation ages using existing luminescence method (SAR OSL) and the appraisal of the recently proposed VSL method to extending the dating range of Brazilian sediments using samples dated using existing OSL methods.

The general observation the work presented in chapter 2, is that the RF intensity differs between quartz from rocks and sediments, and among quartz from sediments with different provenances. The observed linear correlation between OSL, TL sensitivities and UV-RF emission intensity improved the understanding that the sensitization processes in nature might be due to the changes in recombination processes rather than changes in the charge trapping probability. From this correlation, this work also suggested for the first time the use of UV-RF emission intensity for provenance analysis in addition to other provenance proxies. It also supports the suggestion that a single process is involved in sensitization of both TL and OSL. However, further studies are recommended using samples from different provenances in order to confirm the use of the UV-RF signal as provenance proxy.

Chapter 3 investigates the characteristics of VSL signals of quartz from major fluvial systems within Brazil and the use of VSL signals in extending luminescence dating range of Brazilian sediments. VSL protocols used in this study showed that VSL signal of quartz could extend the dating range of fluvial deposits in Brazil especially in the cratonic settings where dose rates are relatively low. Here, it is recommended to use more samples as we observed the dependence of VSL signal on samples. In chapter 4, the OSL dating method was found to be feasible in dating ironstones deposits and the OSL ages are between ~60 ka and 3 ka. The estimated OSL ages of quartz improve the understanding of the genesis of ironstones and suggest that they are related with the Quaternary hydrology of the Xingu River. We recommend the use of single grain approach to determine the time of specific grain trapping, minimize the averaging effect and investigate the possible source of high overdispersion observed for same samples

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