Radiation Measurements 189 (2025) 107543

Contents lists available at ScienceDirect

Radiation Measurements

journal homepage: www.elsevier.com/locate/radmeas

Exploring the correlation between thermoluminescence and optically stimulated luminescence of rose quartz

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ARTICLE INFO

Keywords:
Quartz
TL
OSL
Correlation

ABSTRACT

This work investigates the correlation between thermoluminescence (TL) and optically stimulated luminescence (OSL) signals in natural rose quartz. Four distinct TL glow peaks were identified at 90 °C (I), 140 °C (II), 215 °C (III), and 336 °C (IV) at a heating rate of 1 °C/s. The effect of blue LED illumination on TL glow peaks was investigated revealing a systematic reduction in the intensity of peaks I-III. Additionally, the influence of the temperature on the OSL decay curve characteristics was analyzed, showing a marked decrease in the OSL signal at temperatures above 100 °C. These results contribute to understanding the thermal and optical behavior of rose quartz, with implications for its use in radiation dosimetry.

1. Introduction

In recent years, there has been increasing interest in materials, methods, and processes suitable for retrospective dosimetry, especially in the context of radiation accidents (Bailiff et al., 2016; Chumak, 2013; Göksu, 2003; McKeever and Sholom, 2019; Mesterházy et al., 2012). In this case, it is of extreme importance to quantify the absorbed radiation dose to people and the environment, particularly because dosimeters are not ubiquitously present (Joshi et al., 2021). Various objects can serve for absorbed dose evaluation, including smartphone chip cards, touchscreen glass, electronic components, jewels such as ruby and sapphire, ceramics, and building materials (Bailiff, 1997; Discher et al., 2021; Göksu, 2003; Kumar et al., 2023; Mesterházy et al., 2012). Many of these materials contain quartz, a mineral that is abundant in nature and is widely used in the production of semiconductors, as well as in applications within technology industry, material engineering, and biological and chemical devices (Anas Boussaa et al., 2016; Götze et al., 2005).

Quartz, primarily composed of silicon dioxide (SiO_2), is a significant silica polymorph found in igneous, metamorphic, and sedimentary rocks, with α-quartz being the most common crystalline form in nature (Götze et al., 2021; Williams and Spooner, 2018). This structure is hexagonal, with silicon-oxygen tetrahedral bonds. Brazil holds the largest reserves of quartz crystals globally, followed by Madagascar, Namibia, and China (Rocha, 2015). During the crystallization process, physical, chemical, and thermal processes play a decisive role in the formation, concentration, and nature of structural defects and impurities incorporated into the quartz crystal lattice. These structural imperfections can lead to electronic transitions responsible for luminescence and coloration phenomena in quartz (Kibar et al., 2007; Sharma et al., 2017; Yukihara and Mckeever, 2011). Impurities like Al^{3+}, Ga^{3+}, and Ti^{4+} often substitute Si^{4+} in quartz, and form defects that are critical for luminescence properties (Kibar et al., 2007), including Thermoluminescence (TL) and Optically Stimulated Luminescence (OSL) (Preusser et al., 2009). TL is a phenomenon in which certain materials release stored energy in the form of light when heated, after exposure to ionizing radiation. OSL is a related phenomenon, where the release of the stored energy is triggered by exposure to light (McKeever, 1985; Yukihara and Mckeever, 2011). Both techniques are widely used in dosimetry, particularly for evaluating the absorbed dose in materials following exposure to ionizing radiation (McKeever, 2022; Yukihara and Mckeever, 2011). The use of dosimeters exhibiting both TL and OSL luminescence signal offers several advantages, including (a) enhanced performance under diverse environmental and irradiation conditions, (b) broad applicability across various dosimetric scenarios, (c) improved measurement accuracy. Currently, only two commercial available dosimeters combine TL and OSL capabilities: Al_2O_3:C and BeO (Yukihara et al., 2022). However, numerous studies have investigated alternative materials, such as CaSO4: Tb and CaSO4: Eu (Guckan et al., 2023), MgO: Na, Li (Guckan et al., 2020), quartz (Kitis et al., 2010), among others.

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https://doi.org/10.1016/j.radmeas.2025.107543
Received 7 May 2025; Received in revised form 1 September 2025; Accepted 21 October 2025
Available online 22 October 2025

Recent studies have explored the luminescent properties of the same sample of rose quartz discussed in this work (Chithambo et al., 2025; Ferreira et al., 2024; Martins et al., 2023). TL investigations revealed five distinct glow peaks in rose quartz, at 76 °C (I), 115 °C (II), 180 °C (III), 290 °C (IV) and 423 °C (V) (Ferreira et al., 2024). The first peak (I) appears to be associated with defects related to the $[AlO_4]^-$ and $[X/M^+]^+$ centers (Martins et al., 2023). Furthermore, dosimetry analyses were conducted to evaluate the dose-response, repeatability, reproducibility, and signal fading of the material. The sample exhibited supralinear behavior in the dose range from 0.1 to 1 Gy. The coefficients of variation (C.V.) for repeatability and reproducibility were approximately 1.80 % and 1.30 %, respectively. A rapid fading was observed, with only 12 % of the initial TL signal remaining after 1 h (Martins et al., 2023). Additionally, phototransferred thermoluminescence (PTTL) measurements indicated that peaks I and II exhibited light sensitivity (Ferreira et al., 2024). Recently, Chithambo and collaborators (Chithambo et al., 2025) reported the PTTL in rose quartz using blue, green, and violet light. Violet light reproduced all four peaks, while blue and green light only reproduced the first two (I–II). The PTTL intensity was analyzed using phenomenological and kinetic models, considering systems of donors and acceptors, with the number of donors depending on the preheating temperature. The intensity of PTTL from deep electron traps correlated with the illumination temperature, reflecting combined effects of thermal assistance and thermal quenching at the donor traps and recombination centers.

Based on these previous studies, the present work investigates the correlation between thermoluminescence (TL) and optically stimulated luminescence (OSL) signals in rose quartz pellets. The relationship between TL peaks and OSL in quartz has been examined in several earlier works, particularly with respect to whether both signals originate from the same electron traps (Chruścińska and Szramowski, 2018; Kitis et al., 2010; Kuhns et al., 2000; Wintle and Murray, 1997). One of the earliest studies, by Wintle and Murray (Wintle and Murray, 1997) demonstrated a strong correlation between the OSL signal and a TL peak around 325 °C, showing that optical stimulation empties the same electron traps that would otherwise be thermally released at this temperature. Kuhns and colleagues (Kuhns et al., 2000) reported that the composition of the Linear Modulated Optically Stimulated Luminescence (LM-OSL) signal strongly depends on the quartz source, with different types of quartz exhibiting distinct proportions of luminescence components. Kitis and collaborators (Kitis et al., 2010) further investigated the OSL properties associated with the 110 °C TL peak in natural milky quartz and provided strong evidence that both signals originate from the same electron trap—namely, the 110 °C TL trap and the medium component of the LM–OSL. Thus, no general behavior can be established for quartz underscoring the need for further investigation into TL-OSL correlations.

2. Materials and methods

The natural rose quartz sample was initially crushed into particles smaller than 75 μm. The elemental composition was determined through X-ray fluorescence spectrometry (XRF) using a Malvern Panalytical Zetium XRF spectrometer with standardless calibration (STD-1). To eliminate any pre-existing luminescence signal, the sample was heated at 500 °C for 1 h. The selected powder was then mixed with Durabond 950 binder, a material known to exhibit no intrinsic luminescence signal. To prepare the pellets, 0.310 g of rose quartz powder was combined with 0.090 g of binder, and the mixture was divided into five pellets, each with a mass of $(8.16 \pm 0.79) \times 10^{-2}$ g and thickness of 2.00 $\pm$ 0.5 mm. The mixture was compressed using a 3-ton hydraulic press (SchwingSiwa, model NID 15T/130) to form the pellets, which were subsequently dried at 100 °C for 1 h in a circulating air oven (FABBE, Brazil, mod. 119). Fig. 1 shows the steps of rose quartz pellet production. The trace element composition was analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES) with a Thermo Scientific iCap 6300 Duo. Prior to the measurements, sample grains were fused with lithium tetraborate to ensure complete dissolution. Quantitative analyses were conducted by comparing the results with certified reference materials to ensure accuracy and reliability. Loss on ignition (LOI) tests were conducted at approximately 1027 °C for 2 h to quantify volatile content. Additional ICP-OES analyses were carried out on samples prepared via microwave-assisted acid digestion. This method facilitated the determination of aluminum, calcium, cobalt, iron, potassium, nickel, titanium, and zinc concentrations, using a Horiba Ultima Expert spectrometer for high-precision measurements.

Thermoluminescence (TL) and Optically Stimulated Luminescence (OSL) measurements were performed using a Risø TL/OSL reader (model DA-20). The system is composed of a beta source $^{90}S/^{90}Y$ with a dose rate 10 mGy/s for sample irradiation, blue LEDs for optical stimulation, and Hoya U-340 filter (thickness 7.5 mm; transmission window 290–370 nm) in front of a PMT (EMI 9235QB) for the light measurements. Measurements were performed without a mask to optimize signal detection. To investigate the correlation between TL and OSL two protocols described below were used. This methodology has already been well-established in previous works (Trindade et al., 2019, 2020).

Protocol A: partial OSL.

1. Heating up the sample to 500 °C at a rate of 1 °C/s to empty the traps, then allow it to cool to room temperature (RT)
2. Irradiation to 1 Gy at RT

Fig. 1. Infographic of rose quartz pellets production.

3. Partial OSL measurement with blue illumination during a time interval t_{stop}
4. TL up to 500 °C at 1 °C/s

Steps 2 through 4 were repeated with the illumination time interval t_{stop} in step 3 being gradually increased from 0 to 100 s in 10 s increments. Subsequently illumination time was further increased from 100 to 400 s in 50 s increments. TL results obtained without prior illumination serve as a reference for comparing the signal measured after illumination.

Protocol B: partial TL.

1. Heating up the sample to 500 °C at a rate of 1 °C/s to empty the traps, then allow it to cool to room temperature (RT).
2. Irradiation to 1 Gy at RT
3. Heating up the sample at 1 °C/s till a temperature T_{stop}
4. Cool to room temperature (RT) and CW-OSL measurement during 120 s at RT

Steps 1 through 4 were repeated by increasing the temperature T_{stop} in step 3 from 50 to 500 °C in 10 °C increments. We use an OSL measurement without pre-heating as a reference to compare with the signal obtained after heating.

3. Results and discussion

XRF analysis confirmed that the sample consists of approximately 99.6 % of SiO_2. The ICP-OES analysis provided the average elemental composition of the sample, showing the following concentrations: aluminum (254 mg/kg), calcium (182 mg/kg), iron (164 mg/kg), titanium (23 mg/kg), potassium (14 mg/kg), nickel (2 mg/kg). Trace amounts of cobalt and zinc were also detected, though their concentrations were below the quantification limits of the method. Typically, in quartz crystals, the Si^{4+} ions are substituted by cations such as Al^{3+}, Ga^{3+}, Fe^{3+}, Ge^{4+}, Ti^{4+}, and P^{5+} (Kibar et al., 2007). Some studies suggest that the mechanisms underlying OSL and TL in quartz are strongly associated with structural defects, particularly the $[AlO_4]^-$ and $[X/M^+]^+$ centers (Preusser et al., 2009). The $[AlO_4]^-$ defect is formed when Si^{4+} is replaced by Al^{3+}, requiring positive charge compensation, typically from alkali ions (M^+), hydrogen ions (H^+), or holes (h). The $[X/M^+]^+$ defect involves an impurity (X), such as Ti, that stabilizes in the lattice by bonding with an interstitial alkali ion (M^+).

TL measurements up to 500 °C revealed a prominent peak at 90 °C, identified as the main glow peak, along with additional low-intensity glow peaks centered at 140 °C, 215 °C, and 336 °C. The presence of multiple glow peaks suggests that different charge traps exist in the crystal lattice of rose quartz, each characterized by different trap depths and thermal activation energies and thus releasing charge carriers over specific temperature ranges. Fig. 2 shows the influence of blue LED illumination on the TL glow curve (*protocol A*), plotted on a semi-logarithmic scale to enhance visibility of the less intense peaks. The influence of optical stimulation on TL peaks I, II, and III was evaluated by integrating the area under each glow curve, as shown in Fig. 3. A marked reduction in the intensity of peak I was observed even for short illumination durations, consistent with efficient optical bleaching, typically associated with shallow traps. This result confirms that peak I is dominated by charge carriers residing in low-depth traps that are readily emptied under blue light exposure. In contrast, peaks II and III showed a slower and more gradual reduction in intensity, suggesting that their associated traps are more stable under optical stimulation and require either prolonged exposure or higher stimulation intensity for significant detrapping. These results align with previous observations by (Ferreira et al., 2024), that demonstrated that all four glow peaks in rose quartz are sensitive to blue light, indicating that photo-induced detrapping and phototransferred thermoluminescence (PTTL) process may occur. Such characteristics are particularly advantageous for

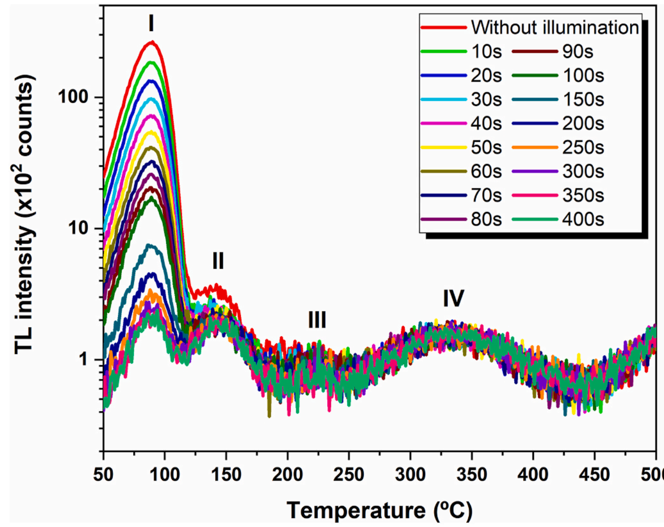

Fig. 2. Influence of previous illumination on the TL glow peaks. A semi-logarithmic scale is used for better visualization.

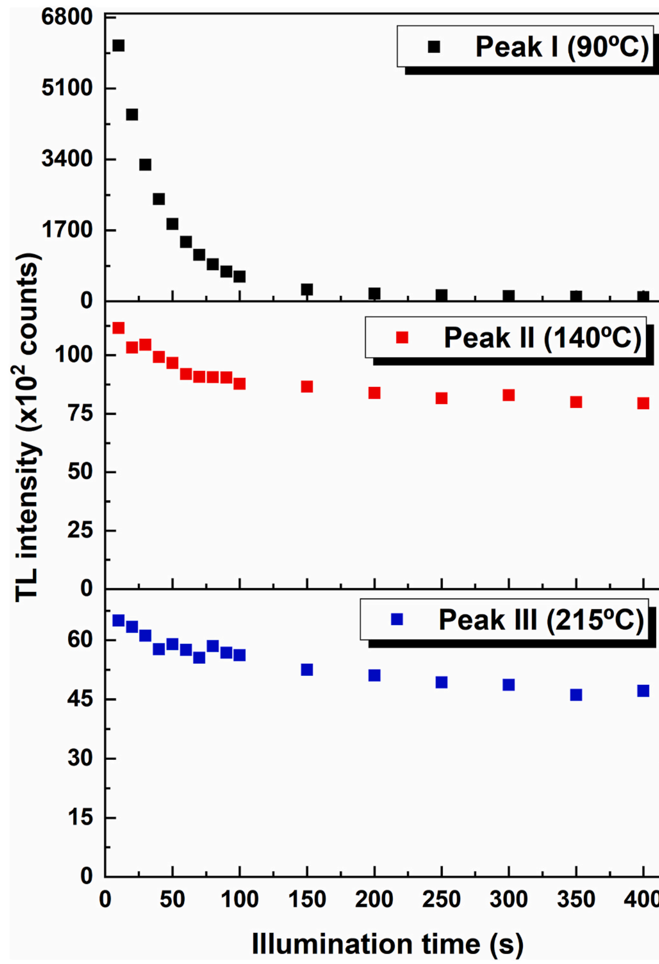

Fig. 3. Influence of previous illumination using blue LEDs on the TL glow peaks. The dots represent the integration of peak I (black solid squares), peak II (red solid squares) and peak III (blue solid squares). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

environmental dosimetry, where materials may be exposed to variable light conditions.

To determine whether the reduction in TL signal is primarily due to optical stimulation (bleaching) or to fading, an additional set of experiments was performed following *protocol A* but omitting the illumination step. Instead, samples were kept in the dark for time intervals equivalent to those used during optical stimulation. As shown in Fig. 4, TL signal degradation due to fading was significantly slower than that observed under continuous blue light exposure. After 500 s of dark storage, the integrated TL signal decreased by approximately 50 %, whereas a similar reduction occurred after only 40 s of blue light illumination. The same behavior was observed in study (Bailey and Holloway, 1997), which showed a reduction in the peak intensity at approximately 110 °C in quartz samples exposed to the OSL excitation light (420–560 nm). These results clearly demonstrate that photo-stimulation leads to a more efficient depletion of charge carriers compared to thermal fading at room temperature (Bailey and Holloway, 1997), reinforcing the strong interaction between optical stimulation and specific TL traps.

The effects of thermal pre-treatment on the OSL signal are shown in Fig. 5, where a semi-logarithmic scale was added to facilitate the interpretation of OSL decay, particularly at low intensities. Heating the sample to 80 °C resulted in a noticeable decrease in the OSL intensity. A more abrupt decline was observed upon heating to 100 °C, where the OSL signal decreased by approximately 80 %. The data suggests that the majority of the OSL signal in rose quartz originates from traps associated with TL peak I, which are shallow enough to be emptied by both thermal and optical stimulation. This dual sensitivity reinforces the hypothesis that the same trapping centers are responsible for both TL and OSL emissions in this material. At temperatures above 100 °C, the OSL signal continues to decay, although more gradually, indicating that deeper traps, associated with higher-temperatures TL peaks, have limited contribution to the OSL response. Further investigations are required to supplement this information.

Fig. 6 summarizes the results of *protocol A*. The red dots represent the integrated OSL signal measured at increasing stimulation times (step 3), while the black dots represent the residual TL measured after illumination (step 4). As expected, the OSL signal increased with longer stimulation time, whereas the residual TL signal exhibited a corresponding decrease. A significant reduction in the TL signal was observed

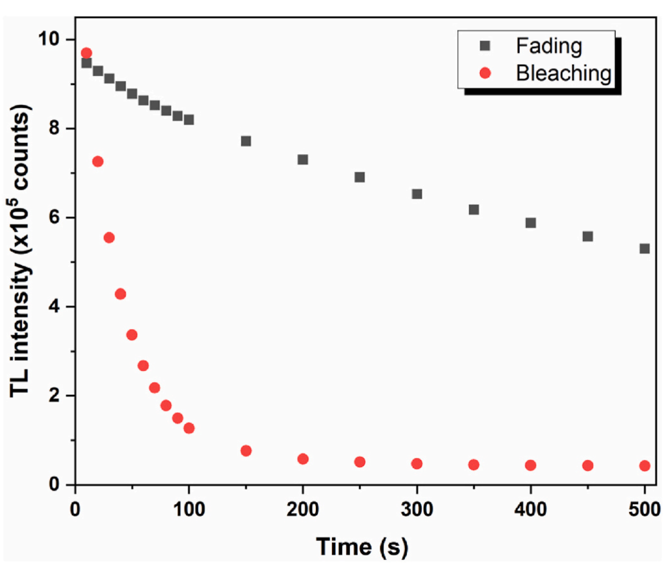

Fig. 4. The integral of the TL curve showing the influence of illumination on the glow peak intensity (red solid circles) in comparation to the effect of pauses in the dark after irradiation on the glow peak intensity (black solid squares). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

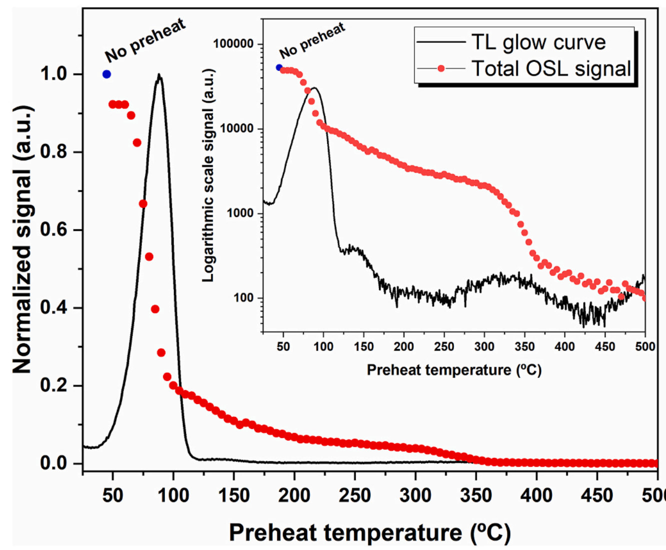

Fig. 5. The influence of step-annealing on the OSL signal. OSL signals were obtained following a series of preheat temperatures from rose quartz samples irradiated with 1 Gy and TL glow peaks exposed to 1 Gy. The red dots represent the integration of the OSL curve obtained at each preheat temperature and the blue dot is the OSL signal without illumination. A logarithmic scale was used for better visualization of the peaks II, III, and IV. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

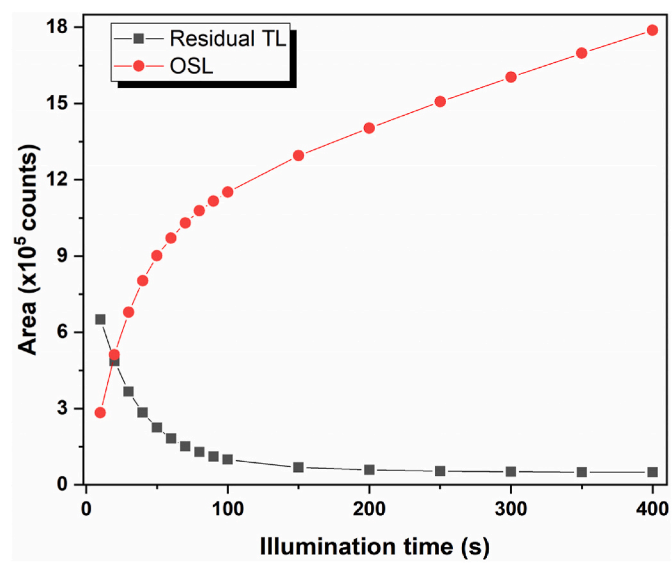

Fig. 6. Integrated residual TL signal from 50 °C to 500 °C (red solid circles) and integrated OSL signal (black solid squares) as a function of the illumination time. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

up to approximately 150s of illumination, beyond which further reduction was minimal. This inverse relationship between the TL and OSL signals suggests that both stimulation modes deplete the same population of trapped charge carriers. A more detailed analysis of the TL glow peaks revealed that peak I intensity declined rapidly with increasing illumination time, while peaks II and III exhibited only minor reductions. To quantify this behavior, the intensities of values of peaks I, II, and III were normalized to their respective non-illuminated TL values. The results showed that the intensity of peak I decreased ~50 % after just 20s of stimulation, while a similar reduction for peak II require approximately 400s. Peak III did not reach the 50 % reduction threshold within the studied illumination range, although its intensity decreased by ~45 % after 400 s. These results provide strong evidence that the OSL signal is predominantly associated with the same shallow trap responsible for TL peak I, confirming a direct correlation between the TL and OSL trapping mechanisms. Additionally, the slight increase in OSL signal observed at extended illumination times is likely attributable to phototransference from deeper traps. This phenomenon further supports the complexity of charge dynamics in rose quartz and highlights its potential for use in dosimetric systems that integrate both TL and OSL readouts.

4. Conclusion

This study explored the correlation between thermoluminescence (TL) and optically stimulated luminescence (OSL) responses in natural rose quartz. A significant reduction in TL peak I was observed even under short illumination times, while peaks II and III exhibited only minor decreases, indicating varying optical sensitivity among the traps. The OSL signal showed a pronounced decline when the sample was preheated to 100 °C, consistent with the thermal release of charge carriers from the same shallow traps responsible for TL peak I. Analysis of the residual TL signal after illumination strongly suggests that the OSL response in rose quartz is predominantly associated with the trap linked to TL peak I, supporting a direct correlation between TL and OSL processes. This overlap in trapping mechanisms reinforces the potential of rose quartz as a dual-mode dosimetric material, capable of providing complementary TL and OSL measurements for radiation dose assessments. Such dual functionality could enhance its applicability in retrospective dosimetry and environmental radiation monitoring.

CRediT authorship contribution statement

I.A. Ferreira: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation. **E.M. Yoshimura:** Writing – review & editing, Validation, Resources, Methodology, Formal analysis. **N.M. Trindade:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This study was financed, in part, by the São Paulo Research Foundation - FAPESP, Brasil (#2025/00094–2; #2024/03006–4; #2022/14925–5, #2021/05042–0, #2018/05982–0). E.M. Yoshimura is grateful to National Council for Scientific and Technological Development - CNPq (#311657/2021–4). N. M. Trindade is grateful to CNPq (#444110/2024-0, #306929/2022–8, #409338/2021–4). The authors thank the Technological Characterization Laboratory (LCT), Brazil, for conducting the chemical analyses.

Data availability

Data will be made available on request.

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