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Optically stimulated luminescence of Durolon polycarbonate

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ABSTRACT

This paper reports for the first time the study of optically stimulated luminescence (OSL) of amorphous Brazilian bisphenol-A polycarbonate (Durolon) exposed to beta radiation. The OSL was studied from two perspectives: characterizing the luminescent signal as a function of radiation dose and examining repeatability. Computational analyses are applied to OSL curves via the deconvolution method. Fluorescence measurements employing the emission - excitation matrix (EEM) technique and optical absorption (OA) are incorporated to characterize the polymer defects and color centers. OSL measurements were carried out in a commercial reader Risø (model DA-20) coupled with a ^{90}Sr/^{90}Y beta source. The sample was illuminated using 470 nm blue LEDs. The OSL response exhibited an exponential decay signal after 1 Gy, with linear dose-response and consistent repeatability. The continuous-wave-OSL curve was well adjusted with two distinct components. As result, Durolon promising potential as a luminescent dosimeter, supported by its desirable OSL signal characteristics.

1. Introduction

Numerous researchers and industries have focused towards on investigating polymer properties due to their low cost, ease of processability, and outstanding mechanical properties [1]. As amorphous and thermoplastic polymer, polycarbonates have attracted considerable attention for their diverse applications. These polymers are utilized as optical data storage devices [2,3] and nuclear track detectors [4]. In addition, they exhibit high optical transmission, and upon exposure to ionizing radiation, their optical density undergoes modification [5]. Within this context, Brazilian Bisphenol-A polycarbonate (Durolon) emerges as a standout polymer, presenting excellent mechanical properties, high transparency, thermal stability, miscibility, processability and anti-weatherability due to its stable amorphous structure [6,7].

Pugliese et al. [8] demonstrated the feasibility of utilizing Durolon as a thermal neutron dosimeter employing track-etch detection methodology. In addition, investigations into alterations in the ultraviolet transmission of polycarbonate under high doses of gamma radiation have been conducted using optical absorbance spectroscopy, nuclear magnetic resonance and positron annihilation [5,9,10]. Additionally, literature findings demonstrate a linear relation between high radiation dose and variations in optical density at specific wavelengths [5]. Although there are published studies on the characterization of this material under neutron and gamma ray irradiation, investigations into the dosimetric characteristics of polycarbonate remain sparse. Hence, beta irradiated Durolon polycarbonate was chosen for investigation using the optically stimulated luminescence (OSL) technique. OSL emission stems from the recombination of charges at luminescence centers, triggered by the absorption of energy by materials previously exposed to ionizing radiation, with its intensity correlating to the absorbed radiation dose [11–13]. The use of OSL as a radiation dosimetry method has grown due to its ability to provide precise and reliable measurements of radiation doses [14].

The investigation of luminescence in organic materials presents inherent complexity owing to the involvement of free radicals generated through diverse mechanisms, which are not intrinsic to the sample itself [15]. In this class of materials such as polymers, radiation-induced process is responsible for the creation of free radicals, which serve as charge traps or recombination centers, facilitating the transfer of electrons and holes to their respective traps [15]. Subsequently, under optical stimulation, recombination events occur, accompanied by light emission, typically detected at a wavelengths shorter than the excitation one to mitigate interference from fluorescence signals [13,14,16].

This study aims to systematically evaluate the dosimetric characteristics of Durolon polycarbonate, focusing specifically on its dose-response behavior and signal repeatability. By elucidating these characteristics, a deeper understanding of the material's suitability for dosimetric applications can be achieved.

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2. Materials and methods

The Durolon® polymer used in this investigation was manufactured by the Brazilian company Unigel Plásticos S/A and distributed by Incomplast [17]. Crystalline phases identification was performed by powder X-Ray Diffraction (XRD) using a EMPYREAN diffractometer with a Cu-Kα radiation source ($\lambda = 1.541$ Å), operating at 40 mA-45 kV, in a 2θ interval between 2.5° and 70° with a 0.026°/min increment. Fluorescence spectra and optical absorption were measured using the Excitation and Emission Matrix (EEM) technique employing a Duetta spectrofluorometer (Horiba Scientific), equipped with a 75 W xenon arc lamp. EEM involves collecting emission scans simultaneously over a range of excitation wavelengths, yielding a two- or three-dimensional graph, providing a comprehensive multidimensional detection of fluorescence compared to other techniques. The first set of measurements covered emission wavelengths between 320 and 370 nm and excitation between 450 and 500 nm (integration time of 2 s and monochromator band pass 10 nm for both emission and excitation). The second set involved emission in the infrared region (830–900 nm) with excitation between 570 and 600 nm (integration time of 15 s and monochromator band pass 10 nm for emission and excitation). Optical absorption data were collected in the range of 300–800 nm, with intervals of 2 nm.

OSL measurements were performed with a Risø TL/OSL reader (DA-20 model). A ^{90}Sr/^{90}Y beta source incorporated in the Risø equipment was used for irradiation, delivering a dose rate of 10 mGy/s, under a nitrogen atmosphere. Luminescence was detected by a photomultiplier tube through a Hoya U-340 filter (7.5 mm) with a transmission window ranging from 270 nm to 380 nm. Illumination was achieved using a set of 470 nm blue LEDs, at room temperature, operated in continuous wave (CW) mode. The measurement procedure involved pre-illuminating the sample to collect the background, irradiating it to a specified dose, and then illuminating it to record the OSL signal. Background was subtracted from the OSL signals for all results.

3. Results and discussions

3.1. X-ray diffraction

Fig. 1 illustrates the X-ray diffraction spectra registered from the pristine Durolon sample. The diffraction pattern of the pristine Durolon sample is characterized by halos extending in the 2θ range from around 5° to 50°. The main diffraction peak, observed as a sharper reflection out of the amorphous part, is centered around 2θ = 17.8°, and suggest the existence of a small crystallized portion in the studied sample [18–20]. The additional peaks observed around 2θ = 29.1°, 30.41° can be attributed to impurities [18–20]. The registered halo profile indicates that the investigated Durolon sample has an predominantly amorphous nature with some degree of internal ordering, evidenced by the sharp reflection at 2θ = 17.8°, which agrees with previous studies for polycarbonates polymers [21].The polycarbonates typically demonstrate slow crystallization kinetics in the absence of additives, owing to their inherent chain flexibility [22–24]. Parameters as solubility, polarity, and hydrogen bonding properties of antisolvents, including dichloromethane utilized in the synthesis of Durolon polycarbonate, exert significant influence on the crystallization behavior of Bisphenol-A [7,22,25].

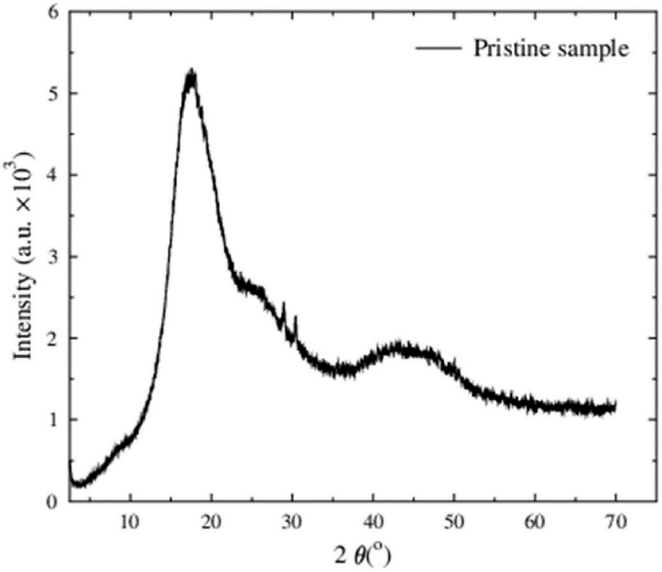


Fig. 1. X-ray diffraction spectra registered from the pristine Durolon sample.

3.2. Emission-excitation spectra

Fig. 2 shows the excitation−emission map registered for excitation ranging from 570 to 600 nm and emission in the near infrared region. The sample presents two emission peaks, a strong emission around 850 nm under excitation lower than 585 nm, and a second emission peak in the range from 850 to 900 nm upon 595 nm excitation. These infrared emissions have been noted by some researchers as potential indicators of photo-oxidation products, associated with oxidation of side-chain and phenyl ring [26]. The phenomenon of photo-oxidation becomes more discernible in subsequent OSL experiments: a degradation in the signal over successive cycles may suggest that prolonged exposure of the material to light induces oxidation.

The UV region, investigated in EEM experiment, was studied due to the luminescent measurement performed by the Risø reader, which provides the observation of a signal in the UV region upon stimulation with blue LEDs (470 nm). To facilitate comparation, measurements were conducted in this wavelength region. Fig. 2 displays a broad emission band ranging from 320 to 370 nm. Previous studies on Bisphenol A polycarbonates using EEM [27] concluded that the emissions observed in the UV region under excitation at 250 nm, with maximum intensity between 300 and 345 nm, are attributable the Bisphenol-A. Additionally, a fluorescence emission at 300 nm, potentially attributed to excimers formed between intermolecular phenyl groups in the polycarbonate main chain, was observed [28]. Others studies have reported the formation of phenyl salicylate bands when polycarbonate is subjected to short irradiation periods and the generation of photo-Fries

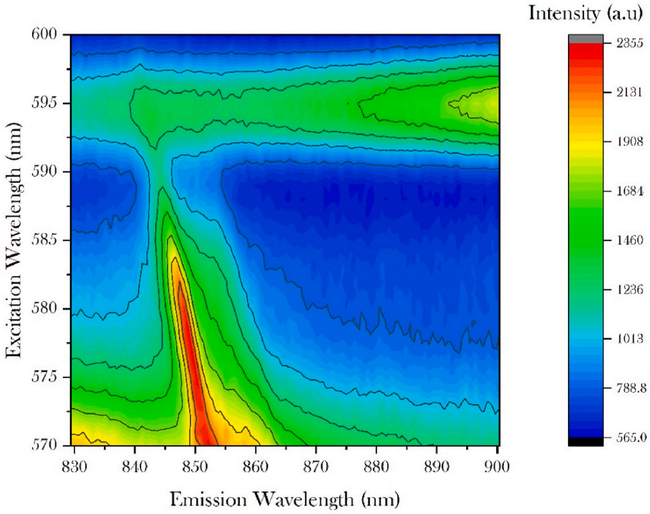


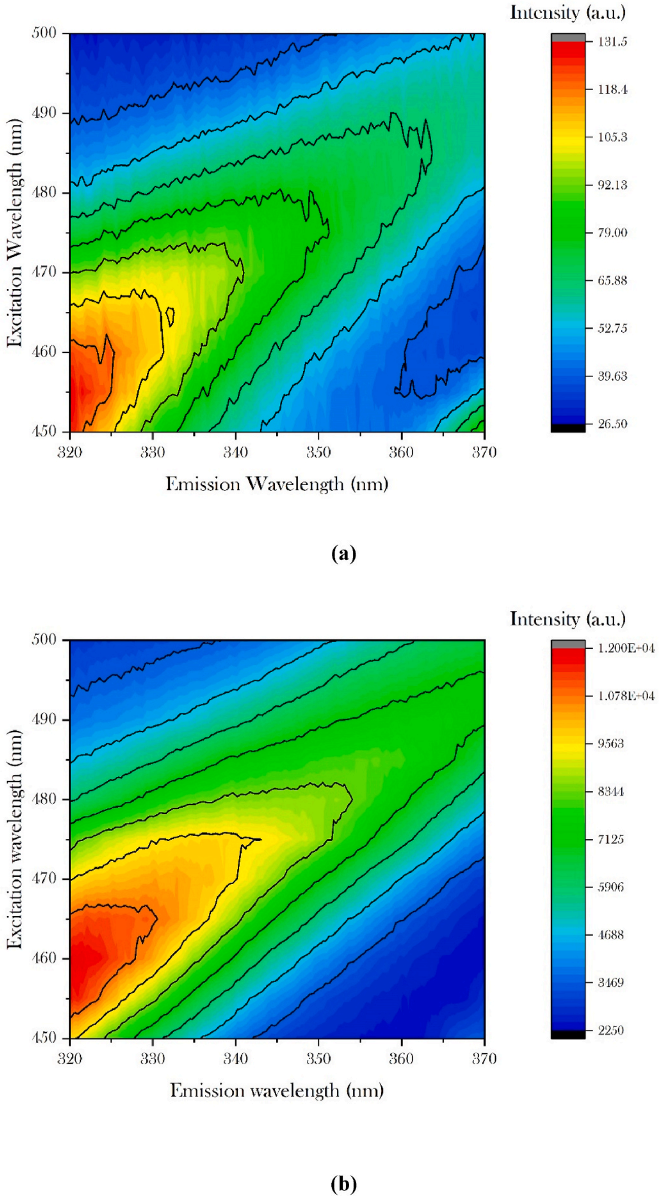
Fig. 2. Durolon Fluorescence Excitation-Emission Spectrum for excitation between 570 and 600 nm and emission in the near infrared region. Integration time 15 s; excitation and emission band pass 10 nm.

rearrangement products during degradation [29,30].

In Fig. 3 the material was excited with photons with a lower energy than the emitted ones, and the Durolon sample was irradiated to verify any changes in the UV signal due to the exposure to beta particles radiation. The registered results obtained using the same parameters for the non-irradiated sample demonstrate no increase in signal or significant alteration post-irradiation. Consequently, the signal observed in the EEM experiment is probably due to intrinsic defects in the polycarbonate structure formed during its formation, rather than as a consequence of beta irradiation.

3.3. Optical absorption

Fig. 4 shows the optical absorption spectrum of pristine and an irradiated Durolon samples, subjected to a 20 Gy beta radiation dose, recorded from 300 nm to 800 nm. The results show a broad absorption band in the ultraviolet range (300 nm–400 nm), corresponding to prominent absorption peaks characteristic of Durolon, consistent with results reported in other studies [31]. The absorption bands centered at 310 nm and 350 nm could be assigned to electronic transitions of π→π* and n →π*, where n stands for nonbonding, related to unsaturated centers of the molecules such as bonds of the carbonyl group (C═O) bonds and C═C bonds [31–33].



(a)

(b)

Fig. 3. (a) non-irradiated and (b) irradiated Durolon Fluorescence Excitation-Emission Spectrum for excitation between 450 and 500 nm and emission in the ultraviolet region. Integration time used was 2 s and the excitation and emission band pass were 10 nm. The sample was irradiated to 20 Gy.

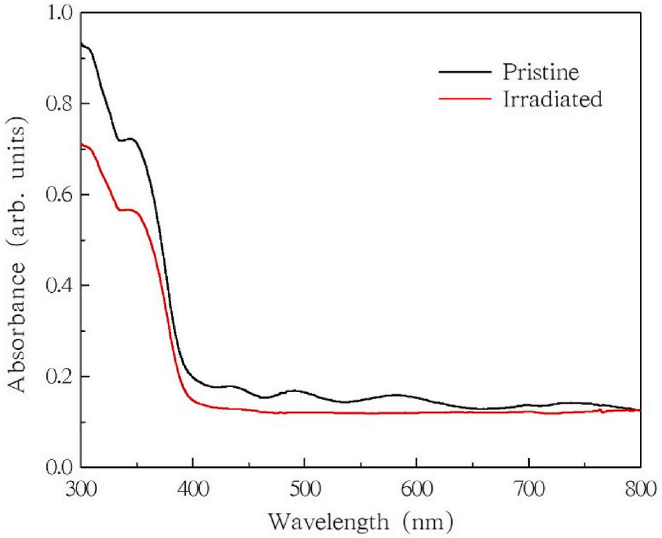


Fig. 4. Optical absorption of pristine and irradiated Durolon samples. The sample was irradiated to 20 Gy.

In the visible range (400 nm–800 nm), the Durolon sample appears transparent, demonstrating transmittance exceeding 70 %. The optical absorbance profile from the pristine sample exhibits some low-intensity absorption bands in this region, which can be induced by adsorbed water and/or additives that prevent degradation due to exposure to sunlight, humidity, and oxygen [22,24,25]. These low-intensity bands in the visible range disappear in the irradiated sample, suggesting that the irradiation induces modification of the water, additives or impurity molecules. The band gap was calculated using the Tauc method for direct transitions [34], a technique widely employed in polymer studies, including polycarbonates [35–37]. The calculated band-gap energies for the Durolon sample are 3.14 ± 0.03 eV for the pristine sample and 3.14 ± 0.04 eV for the irradiated sheet, in agreement with the values published in the literature for polycarbonate [38]. Furthermore, the band gap of the Durolon sample was also determined from the analysis of the spectral dependence of the absorption near the absorption edge. The band gap was calculated using the absorption edge through the determination of the λ_{onset} [39]. The calculated band-gap energies are 3.08 ± 0.04 eV for the pristine Durolon sample and 3.08 ± 0.03 eV for the irradiated sample, in agreement with the values determined by the Tauc method. The determined band gap values suggest that the electrical and optical properties of the Durolon samples remain unaffected after exposure to 20 Gy of beta radiation, in agreement with the EEM results.

3.4. OSL

3.4.1. Preliminary results

OSL measurements were performed to determine the response of Durolon to beta radiation doses. To ensure that we are indeed measuring stimulated luminescence rather than other luminescent processes, various measurements were undertaken with the photomultiplier tube (PMT) on:

- Irradiating the sample and measuring the luminescent signal (conventional CW-OSL).

- Irradiating the sample and checking whether the luminescent signal occurs with Blue LEDs turned off.
- Checking the luminescent signal of a non-irradiated sample with Blue LEDs turned on.

All irradiations were executed using a 5 Gy radiation dose. The results are presented in Fig. 5. The black line corresponds to the signal without illumination after irradiation. It is possible to observe that no signal beyond the background signal was recorded without stimulus. The red dots represent the signal emitted by the non-irradiated sample during illumination. The observed increase in signal relative to the black dots corresponds to its fluorescence, as detected in the same region by the EEM experiment (Fig. 3). However, upon irradiating Durolon and recording its OSL conventional signal, an increase in the initial signal is noticed (blue dots in Fig. 5). This increase stems from the absorption of ionizing radiation by the material, and the consequent trapping of charged carriers in suitable traps. As outlined in the introduction, ionizing radiation typically plays a dual role in materials, generating free radicals that serve as charge traps and luminescent centers, while facilitating the recombination of electrons and holes. In polymers, these electron traps often manifest as dielectric cavity traps, wherein irregular spaces delineated by molecular chains, neutral molecules exhibiting positive electron affinity, unsaturated bonds, and free radicals serve as potential sites [40–42]. However, despite expectations, fluorescence experiments conducted on Durolon following irradiation did not yield any discernible changes in the spectrum. This observation leads us to infer that, within the dose range investigated in this study, ionizing radiation may induce the formation of a low concentration of free radicals, which is not enough to generate significant changes in the fluorescence spectra. According to Chitambo and Kalita (2018) [43], the increase in the OSL intensity is related to a corresponding increase in the concentration of free radicals induced in the irradiation process. Thus, the registered OSL signal may be attributed to intrinsic defects and free radicals within the material that persist despite the recombination process, or possibly to some molecular organization phenomena yet to be fully elucidated. In addition, the recombination process does not extinguish the luminescence centers [15,44]. Consequently, the final signal measured in OSL experiments with this sample is due to photoluminescence and to the radio-induced photo stimulated luminescence. As our sole interest lies in measuring the radio-induced photo stimulated signal of Durolon, subsequent experiments involved subtracting the background signal – the signal of the non-irradiated sample.

3.4.2. Dose-response

To examine the behavior of the OSL signal as a function of radiation dose, the dose-response experiment ranging from 1 to 10 Gy was carried out. The resulting curves are plotted in Fig. 6. The fast exponential decay of the OSL signal was also observed for other polymers, such as ultra-high molecular weight polyethylene, where the luminescence intensity promptly decreases to the background level within the first 5 s of illumination with blue LEDs [43]. At all doses, a fast exponential decay of the signal with the illumination time was observed. Also, despite increasing the irradiation dose applied to the sample, the shape of the decay curve does not change. In contrast to findings reported in literature using high doses of gamma radiation [6,7,45], the sample did not exhibit a yellowish appearance nor did it lose its transparency with an increasing dose, indicating the absence of chain scission or degradation caused by irradiation at this dose level. To verify the variation of initial intensity with the radiation dose, the maximum intensity was plotted as a function of the dose. The inset in Fig. 6 shows a linear relationship between the OSL intensity and the radiation dose. The linear fit applied to the experimental data yields R-square value of 0.999, indicating that the Durolon sample demonstrates a linear dose-response relationship within the range of doses investigated in this work. The observation of the linear dose-response in this work is in full agreement with OSL results for polyethylene polymers also irradiated with beta radiation [43].

3.4.3. Deconvolution of OSL decay curve

The deconvolution of CW-OSL decay curves was done to gain deeper insights into the material and determine the photoionization cross-section (σ). Firstly, the OSL decay curve corresponding to a 10 Gy dose was selected for fitting using OriginLab and Luminescence package in RStudio (Nonlinear Least Squares Fit for CW-OSL curves function) [46]. The best fit was achieved with a model comprising two exponential functions described by equation (1) for the intensity I at stimulation time *t*:

$$I(t) = I_{01}\lambda_1 e^{-\lambda_1 t} + I_{02}\lambda_2 e^{-\lambda_2 t} \tag{1}$$

where I_{0i} is the initial intensity, which is proportional to the concentration of trapped electrons in trap *i*, λ_i are proportional to the detrapping probability [46,47]. The values of I_{0i} and λ_i were determined

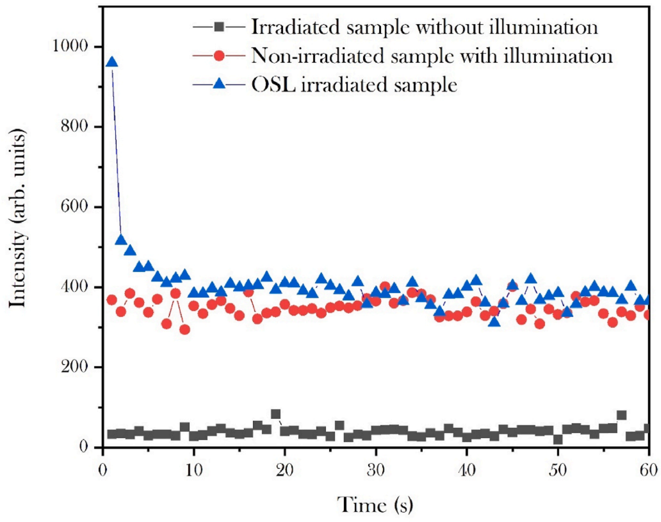


Fig. 5. Comparative OSL decay curves of the irradiated Durolon sample without illumination (black dots) and illuminated (blue dots). The red dots are the signal emitted by a non-irradiated sample during illuminating. The irradiation dose was 5 Gy.

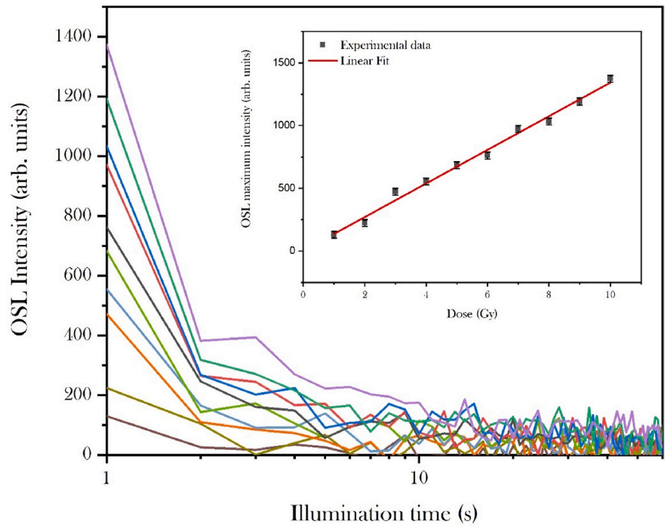


Fig. 6. OSL decay curves varying irradiation dose (1–10 Gy) for Durolon sample. The inset shows the maximum intensity as a function of the dose. A linear function was fitted to the data and is shown ($R^2 = 0.999$). The x-axis is on a logarithmic scale.

iteratively, adjusting to minimize the best Chi-square value. The result is presented in Fig. 7. The exponential curve fits well with the following parameters:

- For the fast component (C1): $I_{01} = (3.4 \pm 0.2) \times 10^3\ \mathrm{m}^{-3}$, $\lambda_{01} = 1.4 \pm 0.2\ \mathrm{s}^{-1}$, and $\sigma = 9.61 \times 10^{-20}\ \mathrm{cm}^2$,
- For the slow component (C2): $I_{02} = (9.5 \pm 1.1) \times 10^3\ \mathrm{m}^{-3}$ and $\lambda_2 = (2.1 \pm 0.4) \times 10^{-2}\ \mathrm{s}^{-1}$, and $\sigma = 6.72 \times 10^{-18}\ \mathrm{cm}^2$.

It is important to note that σ is not a well-defined parameter in complex materials due to the issue of defining the initial and final wave functions of transitions probability [13]. The software automatically provides this value by relating the adjusted λ with the photoionization cross-section using the formula $\lambda = \Phi\sigma$ where Φ is the photon flux.

3.4.4. Repeatability

An important aspect to consider when studying luminescent detectors is possibility of reusing the same sample for various dose determination experiments. The repeatability of measurements was evaluated by executing 26 cycles, each using a dose of 10 Gy, to verify if there were any changes in intensity or if the exponential decay curve was repeated, thus exploring the potential for sample reuse. As shown in Fig. 8, the area under the OSL curves exhibited no significant changes over the course of the 26 runs, with an average of 2091 and a standard deviation of 174, giving rise to a coefficient of variation of 8.0 %. It is likely that this repeatability can be further improved by subjecting the sample to a prior thermal treatment.

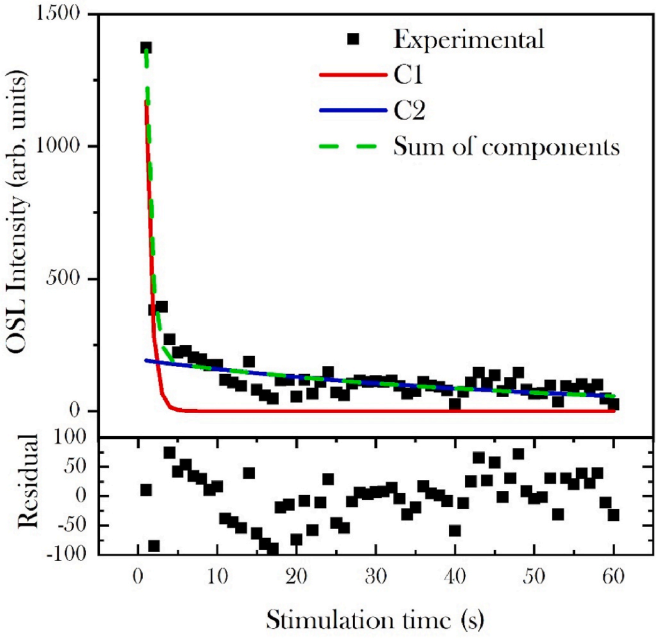


Fig. 7. Deconvolution of the CW-OSL curve of Durolon sample irradiated with 10 Gy, with 2 exponential decay components: C1 – fast component (solid red line), C2 – slow component (solid blue line). The black dots represent the experimental results, and the green dashed line is the sum of both components.

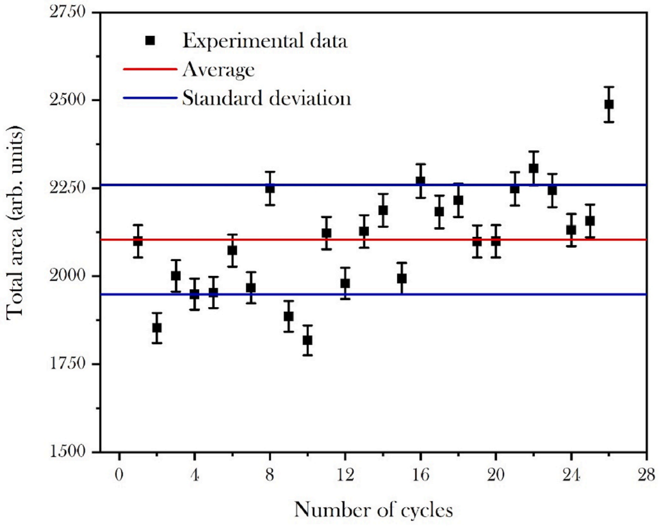


Fig. 8. Area under the curve for 26 cycles of OSL measurements. The error bar was determined by Poisson uncertainty, with the red line representing the average and blue line representing the standard deviation of all measurements.

4. Conclusions

In this work, luminescence and optical investigations were performed on Durolon polycarbonate. Using the EEM technique, strong signals were observed in the UV and IR regions, potentially indicative of oxidation phenomena and photoproducts within polymer's main chain. Various bands were detected in optical absorption spectra, attributed to unsaturated centers within molecules. OSL signal was obtained for beta irradiated samples, exhibiting a linear increase with the irradiation dose in the range from 1 to 10 Gy. By fitting the OSL signal into two components, labeled as slow and fast, photoionization cross-sections of $6.72 \times 10^{-18}\ \mathrm{cm}^2$ and $9.61 \times 10^{-20}\ \mathrm{cm}^2$ were determined, respectively. Moreover, in the repeatability experiment, there were no discernible variations in curve shape or signal, indicating no degradation of the sample irradiated more than 20 times to 10 Gy dose. In conclusion, Durolon demonstrates promising characteristics to act as an ionizing radiation OSL detector.

CRediT authorship contribution statement

A.O. Silva: Writing – review & editing, Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation. **R. Kunzel:** Resources, Investigation, Formal analysis, Data curation. **E. M. Yoshimura:** Writing – review & editing, Validation, Resources, Methodology, Formal analysis. **K.C.C. Pires:** Writing – original draft, Validation, Resources, Formal analysis. **N.M. Trindade:** Writing – review & editing, Validation, Resources, Project administration, Methodology, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Neilo Marcos Trindade reports financial support was provided by Sao Paulo Research Foundation. Neilo Marcos Trindade reports financial support was provided by National Council for Scientific and Technological Development. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgments

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