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Evaluation of the thermally and optically stimulated response of an Italian Obsidian irradiated in ^{60}Co beams

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ABSTRACT

From Obsidian silicate, in its natural form, pellets were manufactured adding Teflon to the material in powder. These pellets were exposed to the gamma radiation beams of a ^{60}Co source in a dose interval from 10 Gy to 10 kGy. The Obsidian in powder was investigated in relation to its physical and chemical characteristics, by means of the X-ray diffraction, scanning electronic microscopy and energy-dispersive X-ray spectroscopy techniques. After the irradiation procedure, the signal of the samples was evaluated by means of three techniques: thermoluminescence (TL), optically stimulated luminescence (OSL) and thermally stimulated exoelectron emission (TSEE). The objective of this work was to verify the TL, OSL and TSEE response of the Obsidian + Teflon pellets after exposure to high doses of gamma radiation. Different response characterization tests were performed, as TL and TSEE emission curves, OSL signal decay, reproducibility, dose-response curves, lower detection limits and fading. From these results, it can be observed that the Obsidian + Teflon pellets present good conditions to be used as high dose gamma radiation detectors; the results obtained with the OSL technique were the most adequate in terms of response in function of absorbed dose. The fading of the signals, varying from about 26% (TSEE) to 44% (OSL), in a time interval of 360 h post-irradiation, also showed favorable results for the use of this material as radiation detector, when the measurements are taken after a defined time interval.

1. Introduction

The Obsidian is a natural material of the silicate family; it is known as a natural glass, and it was first discovered by the Italian Obsius. This stone can be mainly found in the colors brown, black or grey, and, more rarely, in the green, blue or red ones. Obsidian can usually be found in some countries, as Italy, United States of America, Ecuador, Mexico and Japan, and also the Mediterranean Sea. This stone is a volcaniclastic product (Hall, 1997).

Over the years, several reports about studies with different materials from stones were published in the literature, for example, Jasper and Opal, which were already investigated in relation to their luminescent response after exposure to radiation sources (Rocha et al., 2002; Antonio et al., 2016).

The techniques based in the luminescence phenomena as thermoluminescence (TL) and optically stimulated luminescence (OSL), and the thermally stimulated exoelectron emission (TSEE), which is characterized by the removal of electrons of the material surface, are commonly employed to evaluate the signal from irradiated samples, in form of light or electrons, after the exposure of the material to ionizing or non-ionizing radiation beams, and after stimulation by means of heat (TL and TSEE) or light (OSL). The physical processes involved in TL, TSEE and OSL emission are based on the theory of bands in solid state materials.

The choice of the material and the technique to be used depends on the application and of the objective of each study. Nowadays, the radiation dosimetry has covered a large range of applications: medical, space, industrial and in irradiator facilities. This last case involves irradiation facilities which apply doses of radiation for different uses, as inhibition of germination of foods, for example, potatoes, onions and garlic, and disinfestation of grains and citrus fruits (1 Gy to 1 kGy) – "low doses"), water purification and preservation of foods, for example, strawberry, papaya and banana (1 kGy to 10 kGy – "medium doses"), and modification in the color of gems and sterilization of hospital supplies (10 kGy to 100 kGy – "high doses") (McLaughlin et al., 1989; IAEA, 2005).

Initial studies were already performed using Obsidian samples from Greece and the TL and OSL techniques, in order to verify their use as

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radiation dosimeters (Polymeris et al., 2010). In this case, the samples were exposed to the $^{90}Sr + ^{90}Y$ beta radiation source of the TL/OSL system Risø, model TL/OSL-DA-15, and the interval of absorbed dose studied was from 1 Gy to 512 Gy, for TL and OSL techniques.

Göksu and Türetken (1979) presented also results of Obsidian, using the TL technique, showing its good reproducibility of response and shape of its TL glow curve.

Polymeris et al. (2010) obtained a TL glow curve, after irradiation with an absorbed dose of 256 Gy, with peaks, P, at about 90° (P1), 165° (P2), 200° (P3) and 255° (P4). The P1 peak presented a dose response curve with a sublinear response between 1 Gy and 100 Gy; the P2 peak had almost linear behavior between 5 Gy and 500 Gy. The P3 and P4 peaks showed a sublinear response from 10 Gy to 1 kGy. For the study with the OSL technique, the samples were also irradiated with the absorbed doses between 1 Gy and 512 Gy; the results allowed to observe linearity for doses larger than 10 Gy.

This work has the objective of measuring and studying the TL, OSL and TSEE responses of Italian Obsidian + Teflon samples exposed to high doses of a ^{60}Co gamma radiation source, in order to verify the possibility and the feasibility of this material to be used as gamma radiation detector in a range from 10 Gy to 10 kGy. The advantages to use the Obsidian + Teflon pellets are: they present low cost and are reusable (nowadays, the materials used are commercial, as alanine and radiochromic films, and they are expensive). Moreover, there is no reference at the literature about measurements of TL, OSL and TSEE responses of Obsidian + Teflon after exposure to a ^{60}Co source.

2. Materials and methods

A natural stone of black Obsidian (Fig. 1a) was crushed into small pieces and pulverized, using a mortar and a pestle porcelain. This powder passed through two different sieves (sizes of 74 μm and 177 μm), in order to obtain the material in the adequate size (Fig. 1b). The powder obtained was weighed, washed and placed in the drier for about 24 h. After this time, it was mixed with Teflon, using liquid nitrogen to ensure their homogeneity, and then the pellets were manufactured (Fig. 1c), using a uniaxial press with a pressure of approximately 5 kPa, in a proportion of 2:1 of powdered samples of Obsidian:Teflon, with dimensions of 6.0 mm in diameter and 0.8 mm in thickness. After this procedure, these pellets were thermally treated at 350 °C for 30 min and 400 °C for 3 h, in order to assure an adequate uniformity and mechanical strength.

The Obsidian + Teflon pellets were irradiated at the Center for Radiation Technology, IPEN, using the Gamma-Cell 220 System, model 200, Atomic Energy of Canada LTD, with a ^{60}Co source (activity of 47.64 TBq, 10/2014, and absorbed dose rate of 817 Gy/h, September/2016). The time interval between the irradiation and the signal measurement was about 20 min.

All the tests related to the dosimetric properties of the material were analyzed using five TL and OSL samples and nine TSEE samples. The TL and OSL responses were analyzed using the reader system composed by the TL/OSL meter Risø, model TL/OSL-DA-20. The parameters used in the TL measurements were: heating rate of 10 °C/s and a final temperature of 400 °C. In the case of the OSL readings, the conditions were: optical power of 90% for blue LEDs, stimulation time of 80 s, and the use of a filter basket Hoya U-340 in front of the photomultiplier. The TSEE response was obtained using a homemade reader system developed at the Calibration Laboratory (LCI), at IPEN, with a proportional detector (Rocha et al., 2002); the conditions used were: heating rate of 5 °C/s and final temperature of 350 °C. After the TL, OSL and TSEE measurements, the pellets were thermally treated at 400 °C during 1 h, for reutilization.

The physical and chemical characterization of powdered Obsidian was investigated using the X-ray diffraction (XRD), scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDX) techniques. In the case of the XRD technique, a diffractometer Equinox 1000, Inel (with the tube operating in 30 kV/30 mA) was used, and for the SEM and EDX techniques, a scanning electron microscope with an energy dispersive X-ray microanalyser Vega 3 SEM, Tescon, was utilized. In order to make the SEM measurements, the powdered samples were metallized, that is, covered by a thin layer of gold, with the purpose to provide an increase in the electrical conductivity on their surfaces.

3. Results and discussion

Powdered Obsidian was initially investigated in relation to its structure, morphology and chemical composition, by means of the techniques of X-ray diffraction (XRD), scanning electronic microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX), respectively.

The dosimetric characteristics of the Obsidian + Teflon pellets were evaluated in terms of the TL and TSEE emission curves and OSL signal decay curve, reproducibility of response, lower detection limit, variation of the response in function of the dose and signal fading.

3.1. X-ray diffraction (XRD)

The X-ray diffraction pattern obtained for the powdered Obsidian (Fig. 2) shows the formation of broad bands, which is typical of amorphous materials that do not have long-range order. The absence of Bragg peaks confirms the lack of crystalline phases. The result in this experiment demonstrates that the Obsidian is a natural glass; this characteristic was already informed by Polymeris et al. (2010).

3.2. Scanning electron microscope (SEM)

The microstructure of the obtained powder was examined by scanning electron microscopy. Fig. 3a shows a smoothing of the grain surface, confirming the appearance of glass, but with irregular shapes. However, in Fig. 3b it is possible to see a morphology in the form of pointed layers (highlighted). These layers are well visible by the magnification showed at Fig. 3c (highlighted). In addition, it is possible to

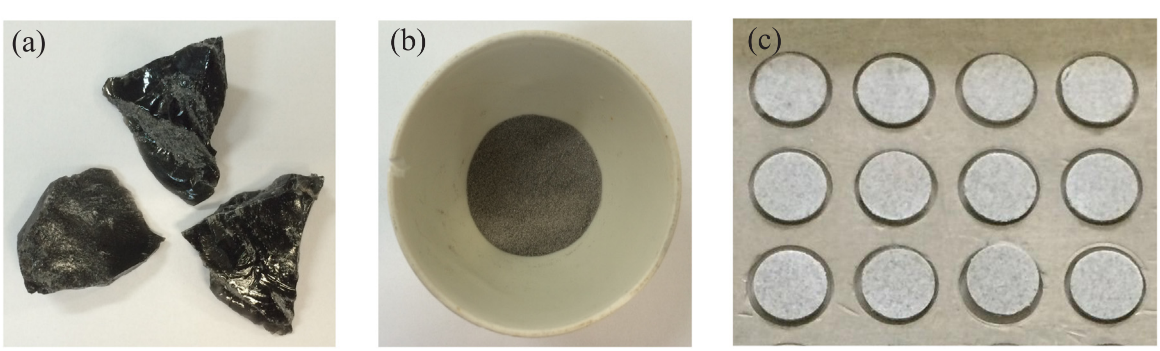

Fig. 1. Italian black Obsidian: (a) in natural form, (b) powder and (c) Obsidian + Teflon pellets.

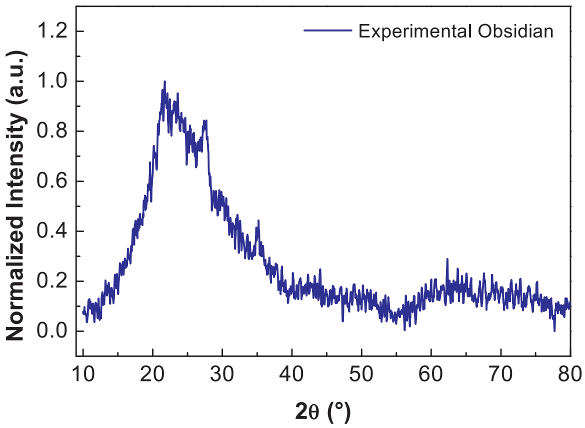

Fig. 2. X-ray diffraction pattern of the powdered Obsidian.

observe in Fig. 3c smaller particles of the material, which possibly were attached on their surface during the metallization performed for the analysis. Polymeris et al. (2010) had already reported that the Obsidian is a silicate.

3.3. Energy dispersive X-ray spectroscopy (EDX)

In order to obtain the chemical composition of the Obsidian, energy dispersive X-ray spectroscopy (EDX) measurements were taken. The results are shown in Table 1. This analysis shows the composition of 82.4% of SiO_2 (predominant presence of amorphous silica) and a variety of other elements, such as Al (7.8%), K (3.8%), Na (3.7%), Fe (1.4%) and Ca (1.0%).

3.4. TL and TSEE emission curves and OSL signal decay

The first studied dosimetric characteristics were the emission curves (TL and TSEE) and signal decay curve (OSL), from measurements taken after the irradiation of the Obsidian + Teflon pellets with an absorbed dose of 1 kGy (^{60}Co). The TL glow curve revealed a dosimetric peak at the temperature of 220 °C. On the other hand, the TSEE emission curve showed a peak at about 300 °C. Fig. 4(a,b,c) presents the curves obtained in this study for the TL, TSEE and OSL techniques, respectively.

Melo et al. (2008) studied four silicates (Actinolite, Tremolite, Rhodonite and Diopside) and obtained a TSEE dosimetric peak around 240 °C, after exposure to an absorbed dose of 20 kGy of ^{60}Co. In the case of this work, the temperature for Obsidian + Teflon was higher. Souza et al. (2000) irradiated samples of Topaz and found TL dosimetric peaks localized at 110 °C, 170 °C and 250 °C; for TSEE, the peak obtained appeared at 190 °C. Jasper samples were studied by Rocha et al. (2002), and a TL dosimetric peak at about 210 °C and a TSEE peak at about 150 °C were presented for an absorbed dose of 2 kGy (^{60}Co).

The results obtained in the present work agree with other of literature in the case of the TL technique. For TSEE, the peak temperature was higher than at the previous works.

Table 1
Chemical composition (%) of the Obsidian samples by the EDX measurements.

Chemical Element	Percentage (%)	
	Weight	Standard Deviation
O	52.6	4.3
Si	29.8	2.9
Al	7.8	1.2
K	3.8	0.7
Na	3.7	1.5
Fe	1.4	0.7
Ca	1.0	0.5

3.5. Reproducibility of TL and OSL responses

The stability of response of the Obsidian + Teflon pellets was verified by means of a reproducibility study. This characteristic was analyzed using TL, OSL and TSEE samples in five cycles of irradiation (absorbed dose of 1 kGy, ^{60}Co), signal evaluation and thermal treatment. The results obtained in the reproducibility test were: 3.0% (TL), 2.9% (TSEE) and 3.1% (OSL), taking into account the average of the group of measurements for each pellet: the mean values were 962.4 counts (TL), 1118.3 counts (TSEE) and 932.3 counts (OSL). After this step, each measurement taken was divided by this average value, and so each measurement was normalized. Fig. 5 shows the response of these normalized measurements in relation to the average value, for the Obsidian + Teflon using the TL, TSEE and OSL techniques.

The use of samples produced by natural materials, as Obsidian + Teflon, presents applications for the purpose of routine work in dosimetry. For this reason, the results obtained can be compared with those established by international recommendations for high-dose dosimeters. According to IAEA (2013), the nominal reproducibility limits for both ceric-cerous sulphate and cellulose dosimeters, which are used as commercial routine dosimeters (ISO, 2002), is 3%. Therefore, it is possible to affirm that the results obtained for Obisidian + Teflon are comparable to the commercial ones, since they varied between 2.9% (TSEE) and 3.1% (OSL).

Other authors already determined the reproducibility of the response for silicates using gamma radiation (^{60}Co). Melo et al. (2008) obtained a reproducibility of the TSEE response in an interval from 12.3% (Tremolite samples) to 18.8% (Diopside samples), for an absorbed dose of 10 Gy.

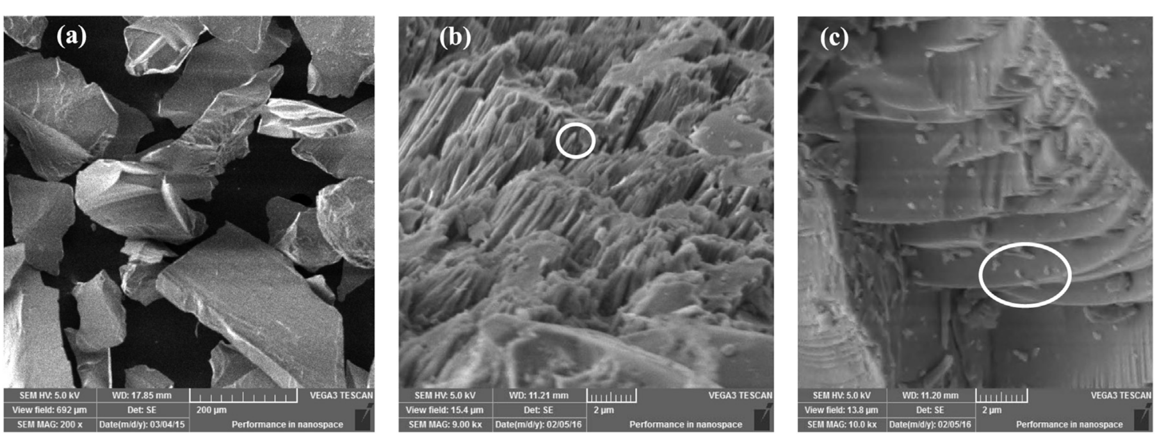

Fig. 3. Micrographs of powdered Obsidian: (a) magnification of 200 × , (b) of 9000 × and (c) of 10,000 × .

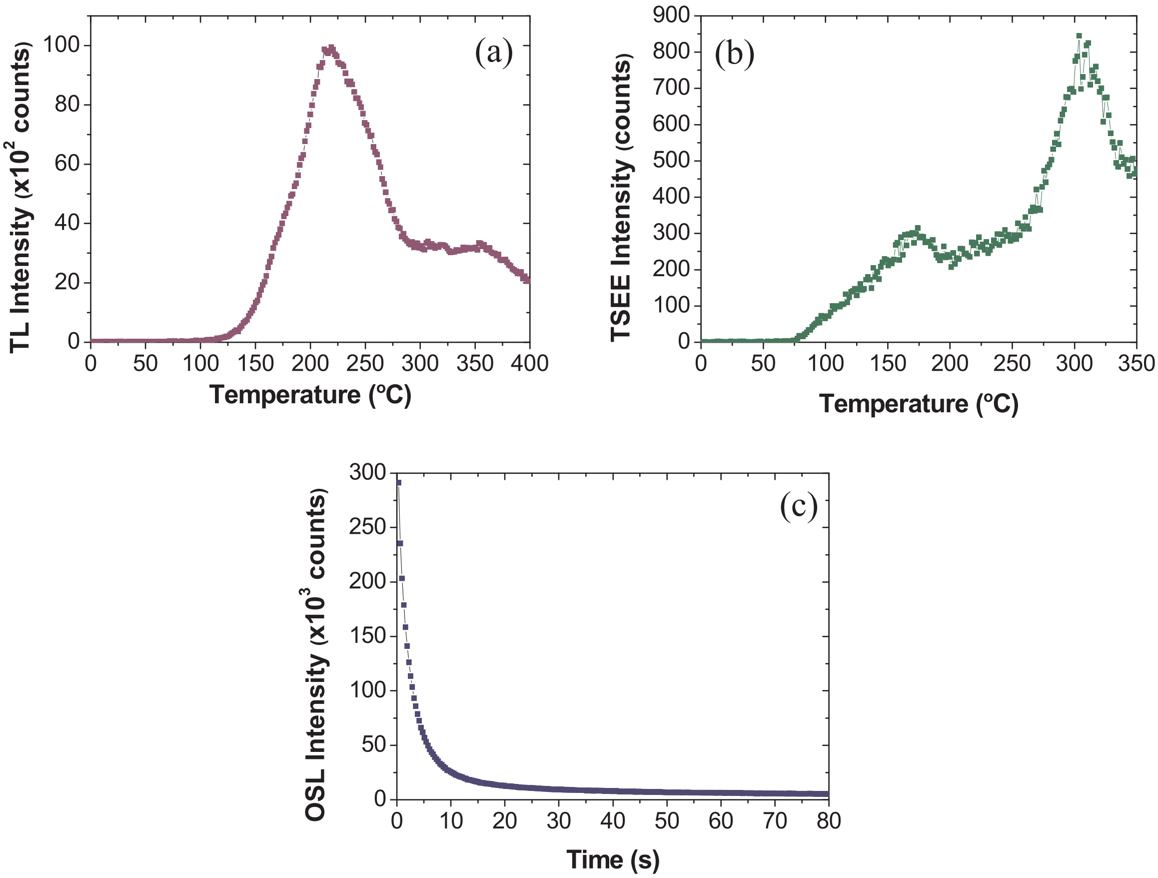

Fig. 4. TL, TSEE and OSL response of the Obsidian + Teflon pellets: (a) TL glow curve, (b) TSEE emission curve and (b) OSL signal decay curve, obtained after an irradiation to 1 kGy (^{60}Co).

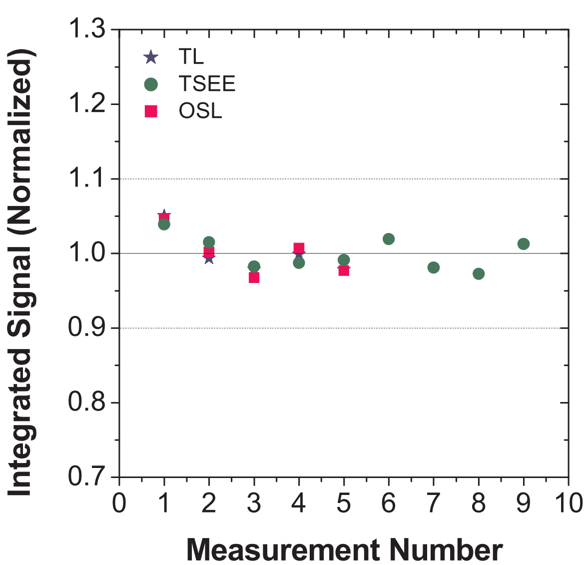

Fig. 5. Reproducibility of the TL, TSEE and OSL responses for Obsidian + Teflon pellets, after irradiation using with a ^{60}Co source (1 kGy).

From the values obtained in the present work, and by comparison with other results already reported in literature, it can be considered that que Obsidian + Teflon pellets presents a good reproducibility of response for all three techniques.

3.6. Lower detection limit (LDL)

Another important characteristic for a dosimeter is its minimum limit of absorbed dose that is detectable, the called minimum detectable dose, or lower detection limit (LDL).

The LDL involves the variation of signal of the samples after their thermal treatment process (measurements of non-irradiated pellets), added to three times the standard deviation of the response of the pellets in the same condition (after thermal treatment), multiplied by a calibration factor (in this case for 1 kGy), obtained for the pellets after irradiation. This method was described by Pagonis et al. (2006). This limit was determined for the three techniques utilized in the present work, and the LDL results were: 48.1 Gy (TL), 18.1 Gy (TSEE) and 79.3 Gy (OSL).

The lower detection limit of the samples was also determined in previous works. Melo et al. (2008) obtained a LDL of 2 Gy for Tremolite and Diopside samples, and using the TSEE technique.

The lower detection limits obtained in this work present a significant difference in comparison to other studied materials. However, the values obtained are acceptable for high-dose dosimetry.

3.7. Dose-response curves

The TL, TSEE and OSL responses of the Obsidian + Teflon pellets can be observed in function of the absorbed dose in an interval from 10 Gy to 10 kGy in Fig. 6. The dose rate was kept the same (0.76 kGy/h) in the case of all the data points.

From the results, it is possible to determine the behavior of the dose-response curves for each technique. In the case of the TL, a supralinearity was observed between only 500 Gy and 2 kGy with a following saturation of the response. In the curve related to the TSEE response, a

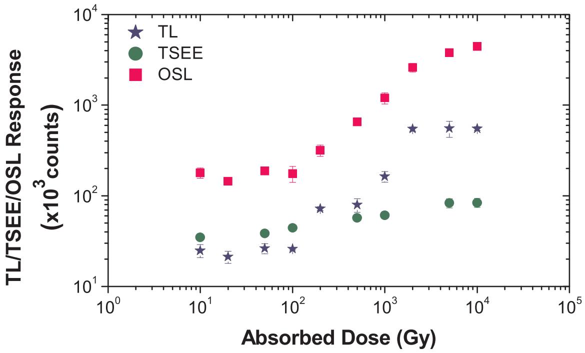

Fig. 6. Dose-response curves for TL, TSEE and OSL techniques, after irradiation of Obsidian + Teflon samples with a ^{60}Co source.

sublinearity was seen (but with an increasing behavior in the whole studied range of absorbed dose), with a following saturation of the response. For the OSL technique, linearity can be observed in the interval from 500 Gy to 2 kGy and then a tendency to saturation. For all three cases, a characteristic of tendency to saturation was obtained.

The literature mentions dose-response curves for other materials irradiated with ^{60}Co beams. Rocha et al. (2002) verified the TL response of sintered Jasper varying the absorbed dose from 10 Gy to 2 kGy, and they observed a linear behavior in the whole studied interval. For the TSEE technique, with doses between 2 kGy and 20 kGy, the result was a tendency to saturation after 10 kGy. Teixeira and Caldas (2014) irradiated different Jasper samples with absorbed doses from 50 Gy to 300 kGy: all five kinds of Jasper samples presented a sublinear behavior between 50 Gy and 1 kGy. Melo et al. (2008) exposed silicate pellets to the absorbed doses from 10 Gy to 20 kGy, and the results showed a sublinear behavior in the whole dose interval.

It is possible to note that the behavior of the dose-response curves can vary according to each silicate material. Thus, the results obtained in this study for Obsidian + Teflon pellets are also acceptable, using calibration factors for their case.

3.8. Fading

The study of the TL, TSEE and OSL signal fading of Obsidian + Teflon pellets was performed after exposure to an absorbed dose of 1 kGy (^{60}Co source). The measurements were taken in specific time intervals after the irradiations: 0 h, 24 h, 48 h, 192 h and 360 h. The behavior of the curves of this study can be seen in Fig. 7. The data points for the different storage times of were obtained for the same pellets.

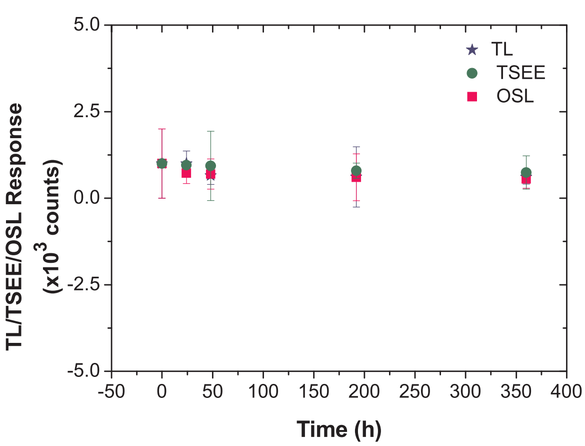

Fig. 7. Fading study of the Obsidian + Teflon pellets after irradiation with a ^{60}Co source, and using the TL, TSEE and OSL techniques.

Table 2
Fading study represented by the remaining signal (in %) of the TL, TSEE and OSL responses of Obsidian + Teflon pellets.

Time (h)	Fading (%)			Remaining Signal (%)		
	TL	TSEE	OSL	TL	TSEE	OSL
0	0	0	0	100	100	100
24	0.03	4.76	27.9	99.9	95.2	72.1
48	34.7	6.69	30.4	65.3	93.3	69.6
192	38.4	21.0	39.7	61.6	79.0	60.3
360	38.7	25.8	44.4	61.3	74.2	55.6

For all three techniques, the maximum standard deviations verified in the measurements were, respectively, from: 3.5% to 10.3% (TL for 360 h and 0 h), 2.8% to 10.8% (TSEE for 192 h and 48 h) and 3.8% to 10.3% (OSL for 24 h and 192 h).

The results of the fading study of the TL, TSEE and OSL techniques can be also observed in terms of remaining signal (in %), as shown in Table 2, for the Obsidian + Teflon pellets.

The fading values mean the signal decay along the time, while the remaining signal represents the amount of signal that still exists after a certain time. In Table 2, it is possible to observe that the signal fades less for the TSEE technique (in relation to the other ones), 360 h post-irradiation, remaining 74.2%. In the case of OSL technique, a greater signal loss occurs (remaining a signal equal to 55.6% after 360 h post-irradiation).

Vila (2012) performed fading studies for some silicates, as Diopside, using TL, TSEE and OSL techniques and a ^{60}Co source (absorbed dose of 1 kGy), for high-dose dosimetry. For the TL response, remaining signals of 85% and 38% occurred after 24 h and 168 h post-irradiation, respectively. In the case of the TSEE response, values of 74% and 34% of remaining signals were also obtained after 24 h and168h post-irradiation, respectively. For the OSL technique, remaining signals of 58% and 30% were obtained in the same time intervals.

The results obtained in the present work are comparable in terms of fading to other silicates.

4. Conclusions

The characteristics of the powdered Obsidian in the point of view of its structure and chemical composition were studied using the X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray Spectroscopy (EDX) techniques. The results proved, basically, that Obsidian is a natural glass and composed mainly of silica (82.4%).

The dosimetric characteristics of the Obsidian + Teflon pellets were investigated by tests of the TL, TSEE and OSL properties. The parameters studied, as TL and TSEE emission curves (dosimetric peaks at the temperatures of 220 °C and 300 °C, respectively) and OSL decay curves, reproducibility of response (with results of 3.0% (TL), 2.9% (TSEE) and 3.1% (OSL)), lower detection limits (48.1 Gy (TL), 18.1 Gy (TSEE) and 79.3 Gy (OSL)) and fading (smaller signal loss for TSEE response and higher for OSL) showed that these samples present interesting results, when compared with other ones presented by different authors, in previous works. In the case of the dose-response curves, the results obtained demonstrated that the Obsidian + Teflon pellets present an appropriate behavior for their use as a high dose dosimeter, especially for the case of OSL technique, which presented the more adequate results.

The pellets investigated in this work were exposed to the absorbed dose range from 10 Gy to 10 kGy, and presented adequate and good results. According to information from literature, it is possible to perform several applications within the studied interval with the Obsidian + Teflon samples. Thus, it is possible to apply this material for different purposes in the agriculture, industry and medical fields, and examples of these applications are disinfestation of fruits and grains and inhibition of germination of foods (1 Gy - 1 kGy), or water purification (1 kGy - 10 kGy), or sterilization of hospital accessories (10 kGy).

In this way, it can be concluded that this material may be considered a promising radiation dosimeter with positive characteristics as low cost, possibility of reutilization and relatively easy manufacturing.

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