OSL properties of $\text{KMgF}_3:\text{Tm}^{3+}$ for dosimetric applications as OSL dosimeterLuis Camargo^a, Lituania Pérez Cruz^b, Epifanio Cruz-Zaragoza^b, Segundo Martínez Ovalle^c, Julián Marcazzó^{a,*}^a Instituto de Física Arroyo Seco (UNCPBA) and CIFICEN (UNCPBA – CICPBA – CONICET), Pinto 399, 7000 Tandil, Argentina^b Instituto de Ciencias Nucleares, Universidad Nacional Autónoma de México, A.P. 70543, 04510 Mexico D.F., Mexico^c Grupo de Física Nuclear Aplicada y Simulación, Universidad Pedagógica y Tecnológica de Colombia, Tunja, Colombia

HIGHLIGHTS

- OSL dosimetric properties of $\text{KMgF}_3:\text{Tm}^{3+}$ were investigated for the first time.
- KMgF_3 doped with 0.5% mol of thulium present the highest OSL efficiency.
- A good linearity in the dose range of 0.1 – 100 Gy was obtained.
- A minimum detectable dose of 0.04 Gy was found.
- The potential of this fluoride as an OSL dosimeter was evaluated.

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ABSTRACT

The optically stimulated luminescence (OSL) properties of undoped and Tm^{3+} -doped KMgF_3 fluoroperovskite have been investigated for the first time. KMgF_3 compounds were synthesized by solid state reaction method and two different procedures were employed to improve the contact among the reagents, namely, the compressed powder was put either in an alumina crucible or in a platinum foil. The maximum OSL emission was found in samples prepared in an alumina crucible and doped with 0.5% mol of thulium. The radioluminescence (RL) spectrum shows two emission peaks at 455 and 360 nm, which can be ascribed to the $^1\text{D}_2\text{-}^3\text{F}_4$ and $^1\text{D}_2\text{-}^3\text{H}_6$ transitions of Tm^{3+} cations. The OSL dosimetric properties of the most promising composition, namely, $\text{KMgF}_3:\text{Tm}^{3+}$ (0.5% mol), have also been determined and analyzed. This compound exhibits good linearity in the dose range from 0.1 up to 100 Gy and satisfactory repeatability with a percentage standard deviation of 2.4%. Therefore, an OSL fading of approximately 75% in the first 36 h of storage is observed and then, the response remains almost constant. These characteristics, together with a minimum detectable dose of 0.04 Gy and the rapid erasing of the OSL signal after 100 s of stimulation, which makes feasible to bleach completely the residual OSL in order to restore the sample between dose measurements, suggest the potential of this perovskite as OSL dosimeter.

1. Introduction

The use of ionizing radiation sources in many fields of everyday life such as radiation therapy, environment monitoring, sterilization or energy generation requires the continuous development of radiation detectors suitable to measure and control doses. Dosimetry based on optically stimulated luminescence (OSL) is a good alternative in environmental and personal dosimetry due to several advantages over others dosimetric methods such as the thermoluminescence (TL). One of such advantages is that the stimulation method is completely optical, which makes simpler the reading process since no heating of the sample

is necessary. For the same reason no thermal quenching occurs and more robust plastics encased OSL dosimeters can be easily manufactured. Moreover, high sensibility of OSL allows multiple readings because it is no necessary long stimulation times and the readout process can be made very fast by increasing the stimulating light intensity (McKeever, 2001).

Only a few numbers of materials are currently used in OSL dosimetry. The most widely used is C-doped alumina ($\text{Al}_2\text{O}_3:\text{C}$) (Perks et al., 2007), which is considered as the standard material for OSL in practical dosimetry. Another one is BeO, which has advantage of being nearly tissue equivalent and shows high efficiency (Sommer et al., 2008).

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However, there is always a constant interest in the search for new materials with improved OSL dosimetry properties.

In the context of the search for new radiation-sensitive materials, pure and doped fluoroperovskite KMgF_3 has been subject of continuous studies due to its good physical properties such as low hygroscopicity, high melting point and intermediate effective atomic number ($Z_{\text{eff}} \approx 14$). Besides, KMgF_3 can easily host many different activators, which enter the lattice by substituting for Mg^{2+} or K^+ . Several studies have been carried out not only from a basic point of view (Gonzalez et al., 2013; Marcazzó et al., 2014) but also by taking into account the possibility of its application as TL or OSL dosimeter (Furetta et al., 1990; Le Masson et al., 2002; Sepulveda et al., 2004; Gonzalez et al., 2006; Marcazzó et al., 2010, Dallas et al., 2010).

In general, it turned out that rare-earth doped KMgF_3 compounds constitute promising candidates to be employed as TL and OSL dosimeters. In particular, it was found that KMgF_3 doped with Eu (Furetta et al., 1990; Gektin, 2000), Pb (Furetta et al., 1990;), Yb (Gambarini et al., 1996), Ce (Le Masson et al., 2002), La (Sepulveda et al., 2004) and Lu (González et al., 2004) have shown to be good candidates for TL dosimetry, being much more efficient than some commercial TL dosimeters (Furetta et al., 1990; Gambarini et al., 1996; Gektin, 2000). On the other hand, the OSL dosimetric properties of this fluoroperovskite doped with Ce has shown high efficiency, even forty times higher than that of $\text{Al}_2\text{O}_3:\text{C}$ (Le Masson et al., 2002). Also, the OSL of Gd-doped fluoroperovskites has been investigated for its use in color center lasers (Somaiah and Narayana, 1993).

Taking into account this background, the aim of this work was to investigate the OSL dosimetric properties of undoped and Tm^{3+} -doped KMgF_3 fluoroperovskite. In particular, the best combination of stimulation light wavelength and filters, the OSL response as function of the synthesis path and dopant concentration, the repeatability of the OSL signal, dose response, the minimum detectable dose and the fading of the OSL signal have been investigated for the first time. Furthermore, in order to evaluate the feasibility of using this fluoroperovskite as an OSL dosimeter, the OSL efficiency of the investigated compound has been compared to that of commercial $\text{Al}_2\text{O}_3:\text{C}$ dosimeter.

2. Materials and methods

The KMgF_3 compounds were synthesized via solid state reaction method and by considering the stoichiometric mixture of the raw materials of pure KF and MgF_2 powders and by following the phase diagram of the binary system KF-MgF₂, (DeVries and Roy, 1953). Thulium was added in aqueous solution ($\text{TmCl}_3 \cdot 6\text{H}_2\text{O}$) in stoichiometric proportions in order to obtain the desired concentrations of the Tm^{3+} impurity in the fluoroperovskite material. In this context, two concentrations of dopant were obtained, namely, 0.2 and 0.5 mol%. Additionally, a reference undoped fluoroperovskite sample was prepared.

The mixtures were compressed to improve the contact between the reagents. Two different procedures were employed to achieve the reaction, namely, the compressed powder was put a) in an alumina crucible or b) in a platinum foil. Then, the powder was inserted into an oven and the temperature was increased from room temperature (RT) up to 700 °C and it kept constant for 5 h. The powder obtained as the product of the reaction was cooled slowly, washed with boiling water and hot ethanol and eventually dried at 200 °C during 2 h. Finally, samples of $\text{KMgF}_3:\text{Tm}$ with grain size between 75 and 250 μm were selected to perform the measurements.

Samples were irradiated at room temperature with a 10 mCi ophthalmic Sr-90 beta-source rendering a dose rate of 0.022 Gy/min at the sample position.

For optical stimulation three different LEDs were used: A Luxeon V Star green LED with maximum emission at 530 nm, a Luxeon V Star blue LED with maximum emission at 470 nm and a Luxeon III Star red LED whit maximum emission at 627 nm. In each case the LED light was filtered by means of two 3 mm thick Schott long-pass filters before

reaching the sample, namely, OG570, OG530 and GG420 long-pass filters for red, green and blue stimulation, respectively. Each long-pass filter features maximum transmission of about 0.9 for wavelengths higher than the cutoff wavelength (570, 530 and 420 nm, respectively) and a transmission less than 10^{-6} at shorter wavelengths.

In order to get rid of the stimulation light, either two 3 mm thick Hoya B-390 or two 3 mm thick Hoya U-340 band-pass filters were interposed between the sample and the light detector. The B-390 filter has non-zero transmission between 320 and 500 nm and maximum transmission (0.77) at 390 nm and the U-340 filter has non-zero transmission between 250 and 390 nm and maximum transmission (0.80) at 340 nm.

OSL signal was detected by means of a photomultiplier tube (PMT) Electron Tube P25PC-02 photon counting head having sensitivity between 180 and 630 nm and maximum response at 350 nm. For all measurements both irradiation and stimulation were applied to the same face of the sample from which the emitted light was detected.

Radioluminescence (RL) spectra were recorded by means of an Acton Research SP-2155 0.150 m monochromator featuring the same PMT aforementioned. Spectra were measured within the wavelength range of 300–800 nm and at a rate of 60 nm min⁻¹.

3. Results and discussion

3.1. Filters and LEDs

Since the OSL dosimetry consists basically in measuring the light emission from a sample previously irradiated while it is being stimulated with light of determined wavelength, knowing the OSL spectrum is crucial in order to determine the optimal combination of filters and LEDs to maximize the collection of emitted light. Because the OSL emission is not stationary, a good alternative is to obtain the radioluminescence (RL) emission spectrum (Cruz-Zaragoza et al., 2017). In general the emission wavelength depends on the recombination centers and it is expectable that the luminescence centers involved in OSL are the same participating in the RL process.

Fig. 1 shows the RL spectra of the KMgF_3 fluoroperovskite undoped (blue line) and doped with 0.5% mol of Thulium (red line). It is possible to see from the figure that samples doped with 0.5% of Thulium emits in two broad bands. The first one is located between 300 and 410 nm and centered at 360 nm and the second one is located between 420 and 500 nm and centered at 455 nm. Both bands centered at 360 and 455 nm can be attributed to the $^1\text{D}_2 \rightarrow ^3\text{H}_6$ and $^1\text{D}_2 \rightarrow ^3\text{F}_4$ transitions of Tm^{3+} ion, respectively (Quintanilla et al., 2011). On the other hand, undoped sample shows a subtle broad band with maximum around 350 nm. This broad emission band at 350 nm has already been observed

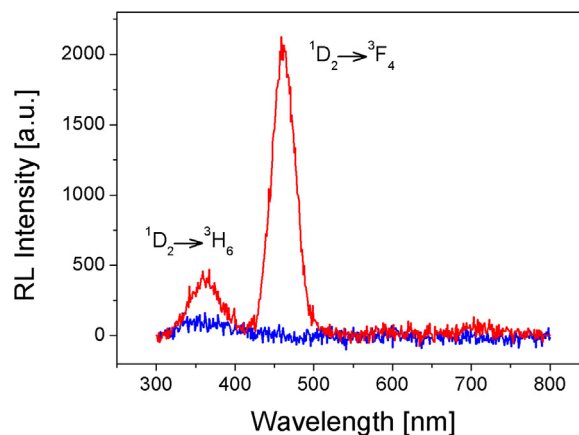


Fig. 1. RL spectra of KMgF_3 fluoroperovskite undoped (blue line) and doped with 0.5% mol of Thulium (red line). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article)

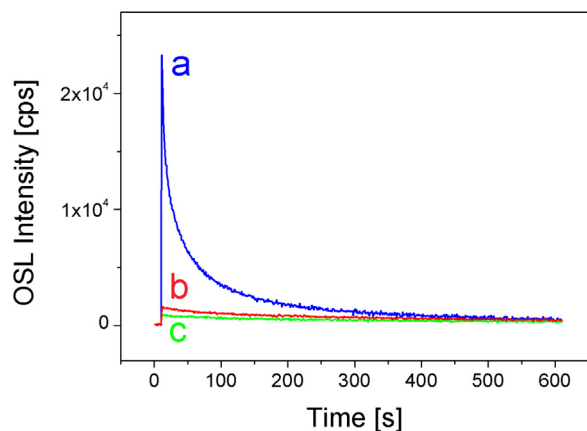


Fig. 2. OSL decay curve of $\text{KMgF}_3:\text{Tm}$ (0.5% mol) under blue (a), red (b) and green (c) light stimulation. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article)

by other authors and it is dominant in the thermoluminescence of KMgF_3 host (Furetta et al., 1990). This peak is attributed to the thermal activation of F centers, created during irradiation.

From the spectra of Fig. 1, two configurations of filters (emission filters) were selected to interpose between the sample and the light detector, i.e., two Hoya B-390 and two Hoya U-340 band-pass filters with transmission between 320 and 500 nm and, 250 and 390 nm, respectively. On the other hand, as it was detailed in the Materials and Methods Section, three light sources for stimulation were selected; namely, red, green and blue light with maximum emissions at 627, 530 and 470 nm, respectively. When the samples were stimulated with blue light, only the configuration with the Hoya U-340 filters was investigated because of the overlapping of the wavelength.

Fig. 2 shows the effects of red, green and blue stimulation light on the OSL response of $\text{KMgF}_3:\text{Tm}$ (0.5% mol) after irradiating the sample with a dose of 2.2 Gy of beta radiation. It is evident from the figure that OSL signal is more intense when blue light is employed. This result has been observed in previous works regarding the OSL of others rare earth doped KMgF_3 , i.e., in the OSL of $\text{KMgF}_3:\text{Ce}^{3+}$ (Le Masson et al., 2002). By taking into account these results, in what follows all OSL measurements have been made with blue stimulation and Hoya U-340 band-pass as emissions filters.

3.2. Influence of the preparation procedure on the OSL response

As it was mentioned above, two different procedures were employed to achieve the reaction, namely, the compressed powder was put either in an alumina crucible or in a platinum foil. In Fig. 3 it is possible to see the OSL decay curves of irradiated $\text{KMgF}_3:\text{Tm}$ (0.5% mol) and undoped KMgF_3 corresponding to each preparation procedure. As it is evident from the Fig. 3, for both $\text{KMgF}_3:\text{Tm}$ (0.5% mol) and undoped KMgF_3 , the highest intensity is obtained when samples are prepared in an alumina crucible. This effect could be related to the fact that alumina allows ion migration to the host of the compound when the mass is being melted at the preparation temperature, which, in turn could increase emission yield. In what follows, we will focus on samples prepared by this procedure.

3.3. OSL response as a function of dopant concentration

In order to determine the amount of doping that yields the highest OSL efficiency of the investigated samples, Fig. 4 shows the OSL curves of samples of KMgF_3 doped with 0.5% and 0.2% mol of thulium and undoped KMgF_3 . All samples were irradiated with a dose of 2.2 Gy and stimulated with blue light.

Whereas that undoped KMgF_3 sample has lowest OSL efficiency

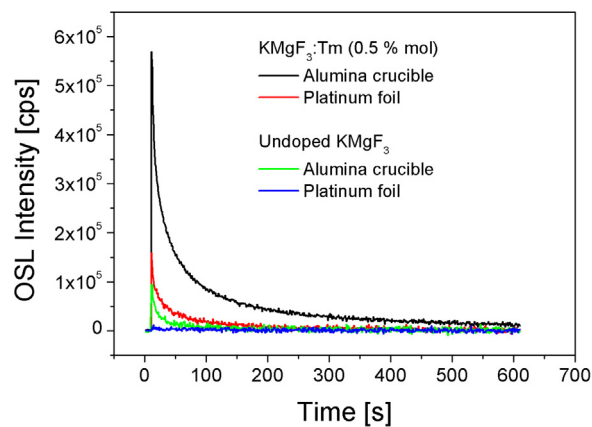


Fig. 3. OSL decay curves of irradiated $\text{KMgF}_3:\text{Tm}$ (0.5% mol) and undoped KMgF_3 fluoroperovskite powder as a function of each preparation procedure.

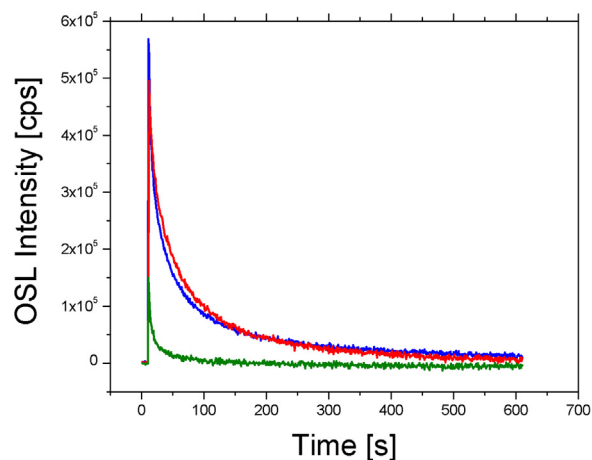


Fig. 4. OSL decay curves of irradiated $\text{KMgF}_3:\text{Tm}$ (0.5% mol) (blue curve), $\text{KMgF}_3:\text{Tm}$ (0.2% mol) (red curve) and undoped KMgF_3 (green curve) fluoroperovskite samples. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article)

(green curve), samples of KMgF_3 doped with 0.2% and 0.5% mol of Tm present almost the same intensity (red and blue curve, respectively). However, of the two concentrations studied in this work, the signal corresponding to the sample doped with 0.5% mol of Tm (blue curve) shows the most intense OSL peak and highest integrated OSL intensity as well. This fact has already been observed in other compounds by several authors (Mishra et al., 2002; Marcazzo et al., 2004, 2009; Cruz-Zaragoza et al., 2016) where for low dopant concentration, the OSL or TL intensity increase with the dopant concentration up to a maximum but then, the signal begins to decrease. In fact, by increasing the concentration of rare earth cations more luminescence centers can be created and hence OSL or TL intensity also increases. However, the glow intensity cannot be expected to increase indefinitely with concentration, since the rate of formation of active luminescent centers by capturing the holes during irradiation might fade rapidly and the so-called concentration quenching effect occurs (Mishra et al., 2002). Nevertheless, it will be necessary to synthesize other percentages of doping in order to determine the optimum concentration.

3.4. Repeatability of the OSL response

One of the characteristics to be taken into account when a material is evaluated as a possible dosimeter is the repeatability of its OSL signal. In general, both the shape and the area under the OSL curve are investigated. Fig. 5 shows the repeatability of the OSL signal of the

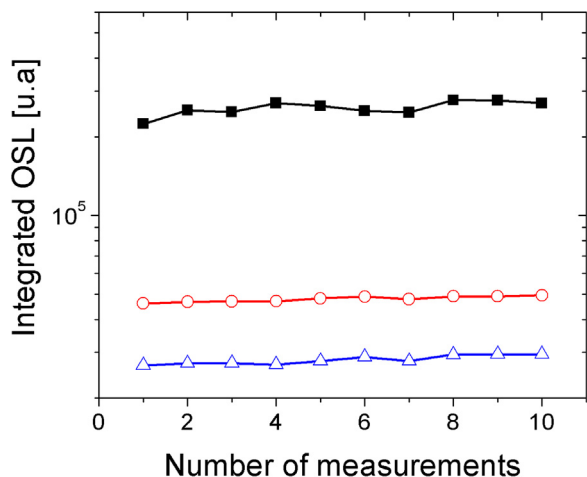


Fig. 5. Repeatability of the OSL response when it is integrated the first 10 (hollow blue triangles), 20 (hollow red circles) and 600 s (filled black squares) of the OSL curve, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article)

fluoroperovskite with highest OSL intensity, namely, $\text{KMgF}_3:\text{Tm}$ (0.5% mol), when different portions of the OSL curve are integrated. It is possible to see a very acceptable repeatability of the OSL signal with a percentage standard deviation of 3.8%, 2.4% and 6.2%, when it is integrated the first 10, 20 and 600 s of the OSL curve, respectively. In all cases, samples were irradiated with a beta radiation dose of 2.2 Gy. On the other hand, the shape of the OSL curves is the same in all the measurements.

3.5. Dose response and minimum detectable dose

Fig. 6 shows the OSL curves of $\text{KMgF}_3:\text{Tm}$ (0.5% mol) fluoroperovskite. The sample was irradiated with different doses of beta radiation, namely, 0.22, 0.66, 2.2, 6.6, 22 and 90.64 Gy, from bottom to top respectively.

Furthermore, Fig. 7 shows the dose response when the OSL signal is integrated along the first 20 s from the beginning of the stimulation. It was selected a time of 20 s for integration interval because this time presented the better repeatability with a percentage standard deviation of 2.4% (see previous section). As it can be seen from Fig. 7, good linearity in the studied dose range was obtained and an adjusted R-square equal to 0.995 was found when a linear regression was performed on the experimental data.

Besides, the minimum detectable dose (MDD) of this fluoroperovskite has been determined by means of $\text{MDD} = 3\sigma_{\text{BG}}$, being σ_{BG} the

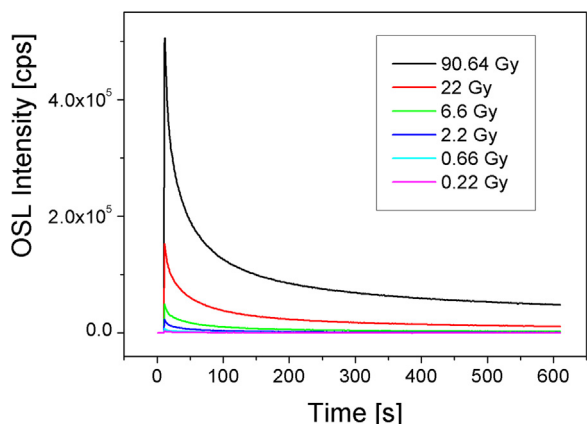


Fig. 6. OSL curves of $\text{KMgF}_3:\text{Tm}$ (0.5% mol) irradiated with different doses, ranging from 0.22 up to 90.64 Gy from bottom to top respectively.

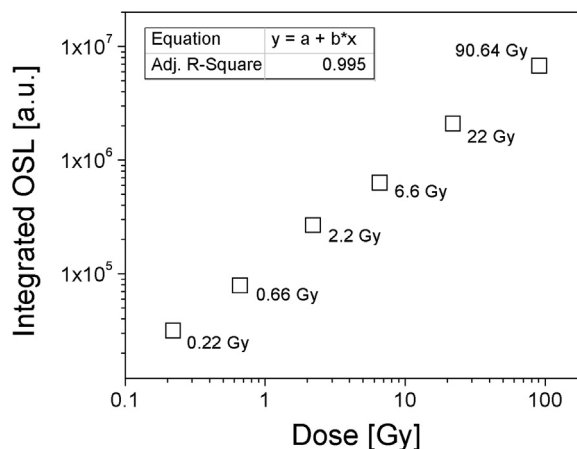


Fig. 7. Dose response $\text{KMgF}_3:\text{Tm}$ (0.5% mol) of when it is integrated the first 20 s of the OSL curve.

experimental standard derivation of the background signal recorded by using blank detectors. These measurements were carried out by using the same samples except that they were not irradiated (Yukihara and McKeever, 2011). For $\text{KMgF}_3:\text{Tm}$ (0.5% mol) a value $\text{MDD} = 0.04$ Gy has been found.

3.6. Fading of the OSL signal

From the point of view of the applications of this compound to OSL dosimetry, it is of interest to investigate the fading of the OSL signal as a function of the time elapsed between irradiation and readout. In this context, sample was irradiated with a dose of 2.2 Gy of beta radiation at room temperature (RT) and storage in darkness and at RT for different periods of time. Fig. 8 shows the integrated OSL signal as a function of several storing intervals, namely, 0.08, 0.16, 0.5, 1.6, 19.2, 72 and 163.5 h. It can be seen from the figure that OSL signal presents an important fading of approximately 75% after the first 36 h of storing and then, the response remains almost constant.

3.7. Comparison with $\text{Al}_2\text{O}_3:\text{C}$

Finally, in Fig. 9 it is possible to see the OSL efficiency of (a) the standard commercial $\text{Al}_2\text{O}_3:\text{C}$ dosimeter (Landauer, Inc.) compared with (b) the $\text{KMgF}_3:\text{Tm}$ (0.5 mol%). Samples were irradiated with a

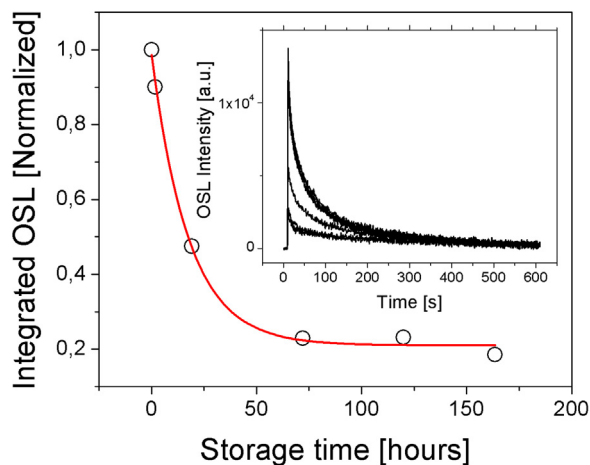


Fig. 8. Integrated OSL signal of $\text{KMgF}_3:\text{Tm}$ (0.5% mol) as a function of the storage time (hollow circles). Continuous line was obtained by fitting by a single exponential. In the inset: the OSL response after 0.08, 0.16, 0.5, 1.6, 19.2, 72 and 163.5 h. of storage, from top to bottom one after another.

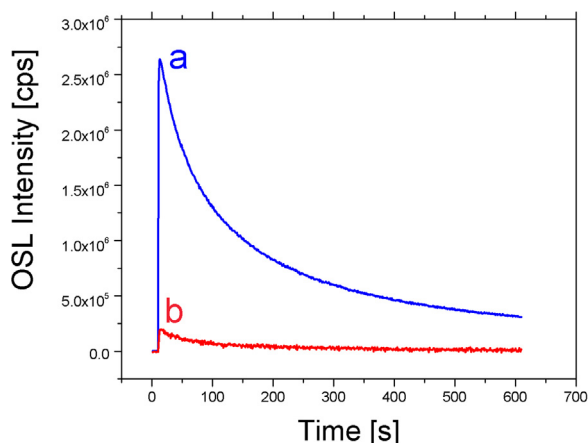


Fig. 9. The OSL decay curve of (a) commercial $\text{Al}_2\text{O}_3\text{:C}$ and (b) $\text{KMgF}_3\text{:Tm}$ (0.5 mol%). Both OSL signals have been recorded under the same experimental conditions and normalized to the sample weight.

beta dose of 2.2 Gy and OSL curves were normalized to the sample weight, i.e., OSL response was divided by the weight of each sample.

As it is evident from the figure, the OSL response of $\text{KMgF}_3\text{:Tm}$ (0.5 mol%) is one order of magnitude lower than that of commercial $\text{Al}_2\text{O}_3\text{:C}$ dosimeter, which in principle could be considered as a disadvantage. However, it is necessary to take into account that the OSL signal of the perovskite quickly fades out after 100 s of stimulation. This could make easier to bleach the residual OSL in order to restore the sample between dose measurements. In the case of $\text{Al}_2\text{O}_3\text{:C}$, the long decay time of its OSL signal could become a drawback if total depletion of traps is necessary before each OSL measurement (Gaza, 2004).

4. Conclusions

The OSL dosimetric properties of the sample of KMgF_3 doped with two concentration of thulium have been investigated for the first time. In particular, it was found that samples prepared in an alumina crucible and doped with 0.5% mol of thulium present the highest OSL efficiency.

The RL spectrum of $\text{KMgF}_3\text{:Tm}^{3+}$ fluoroperovskite shows two broad bands centered at 360 and 455 nm which can be attributed to the $^1\text{D}_2 \rightarrow ^3\text{H}_6$ and $^1\text{D}_2 \rightarrow ^3\text{F}_4$ transitions of Tm^{3+} ion, respectively. On the other hand, undoped sample has an only faint broad band with maximum around 350 nm, which can be attributed to the thermal activation of F centers created in the host during irradiation.

As to the light sources studied for stimulation, the blue light (470 nm) stimulation with the Hoya U-340 filters presented the maximum OSL response.

The best repeatability of the OSL signal is found when the OSL signal is integrated for the first 20 s of the readout. In this case a percentage standard deviation of 2.4% is obtained. Very acceptable linearity in the dose range of 0.1–100 Gy was obtained and a minimum detectable dose of 0.04 Gy has been found. An OSL fading of approximately 75% in the first 36 h of storage is observed and then, the response remains almost constant.

Although it was found that the OSL efficiency of $\text{KMgF}_3\text{:Tm}$ (0.5 mol%) is one order of magnitude lower than the OSL response of commercial $\text{Al}_2\text{O}_3\text{:C}$ dosimeter, the rapid decaying of the OSL signal of the doped perovskite could be taken as an advantage, since it could make easier to bleach the residual OSL in order to restore the sample between dose measurements.

The results of this work show that the investigated Tm-doped fluoroperovskite could be envisaged as new promising OSL dosimeter deserving further investigations.

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