

Thermal Effect on TL Response of Single Doped LiF+NaF:RE Polycrystalline Phosphors

E. GÓMEZ-CHAYRES¹, E. CRUZ-ZARAGOZA^{2*}, M. I. GIL TOLANO^{1,2},
L. PÉREZ CRUZ¹, P. R. GONZÁLEZ³, M. C. SALCEDO LUNA⁴

¹Posgrado en Ciencias Químicas, Facultad de Química, Universidad Nacional Autónoma de México, Av. Universidad 3000, 04510 Ciudad de México, México.

²Instituto de Ciencias Nucleares, Universidad Nacional Autónoma de México, A.P. 70543, 04510 Ciudad de México, México.

³Instituto Nacional de Investigaciones Nucleares, Carretera México-Toluca S/N, 52750 Ocoyoacac, Estado de México, México.

⁴Unidad de Servicios de Apoyo a la Investigación, Facultad de Química, Universidad Nacional Autónoma de México, Av. Universidad 3000, 04510 Ciudad de México, México.

*Email: ecruz@nucleares.unam.mx

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Abstract In this work, the sintering and annealing effects on the thermoluminescent (TL) behavior of undoped and rare earth (RE)-doped LiF+NaF powder samples (RE = Ce³⁺, Eu³⁺, Lu³⁺ or Tl⁺, at 0.5 mol%) was analyzed by evaluating the sensitivity to gamma radiation and TL response of the material. The polycrystalline samples were obtained by solid state reaction at 1000°C. The samples were irradiated in a Gammacell-3000 Elan irradiator loaded with ¹³⁷Cs sources. The glow curves of the LiF+NaF doped with lutetium or thallium show an intense glow peak at about 175°C and 135°C, respectively. When the phosphor was doped with cerium or europium the glow curves were complex in their structure, with TL peaks observed at 155°C and 165°C, respectively. The linear dose-response was between 10 and 50 Gy for cerium, europium or lutetium doped LiF+NaF samples, while for the thallium doped and undoped samples such intervals were 10-100 Gy and 10-500 Gy, respectively. Because the shape of the glow curves were complex, the analysis was carried out in (i) samples without a sintering treatment where the TL response was found insensitive to pre-irradiation annealing treatment, and (ii) sintered samples (300, 350, 400 or 500 °C), in this last case the TL response was dependent on the annealing temperature (100-400 °C), finally (iii) the kinetics parameters of the glow curves were analyzed by assuming a general

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Gómez-Chayres, E. order kinetics model. The observed glow curves and TL characteristics of the
Cruz-Zaragoza, E. LiF+NaF:RE phosphor make attractive this material to be useful in gamma
Gil Tolano, M.I. dose dosimetry.
Pérez Cruz, L.
González, P.R. **Keywords:** thermoluminescence; dosimeters; thermal effect; rare earth;
Salcedo Luna, M.C. alkaline fluorides.

1. INTRODUCTION

Alkaline fluorides have been attractive materials on luminescent applications because of their high sensitivity to ionizing radiation. Lithium fluoride is a well known radiation-sensitive material used in broad applications like photonics, radiation dosimetry and x-ray imaging [10, 12, 1]. Its wide use in optical applications is related to its chemical stability, non hygroscopicity and its ability to host stable electronic defects even at room temperature [11]. On the other hand, sodium fluoride has been use too as TL phosphor with a good sensitivity to beta, gamma and x-ray radiation [3, 8, 13]. The effect of heat treatments on the glow curve and TL response of lithium fluoride had been extensively studied by several authors *e.g.* [2, 4, 5, 7] showing the strong dependence on the thermal history preparation of the samples as well as on the annealing treatments. In this work, the sintering and annealing effects are analyzed for the LiF+NaF:RE (RE = Ce³⁺, Eu³⁺, Lu³⁺ or Tl⁺, at 0.5 mol%) polycrystals because its suitable glow curves structure and the linear dose-response could be used for low dose radiation dosimetry.

2. EXPERIMENTAL PROCEDURE

Undoped and rare earth-doped LiF+NaF polycrystalline samples were prepared by conventional solid state synthesis. All reagents were of analytical grade purity. An equimolar mixture of lithium fluoride and sodium fluoride were placed in a platinum crucible at 400°C and after half an hour the furnace temperature was adjusted to 1000°C. The temperature was then lowered to 450°C and slowly cooled to room temperature (RT). The impurities of rare earth ions (Ce³⁺, Eu³⁺, Lu³⁺ or Tl⁺) were incorporated to the melt as the necessary volume (to reach 0.5 mol% concentration) of the chloride solution. The wet mass was dried at 100°C during twelve hours. Powder X-ray diffraction (XRD) patterns were recorded in a D8 Advanced Davinvi Bruker® diffractometer using Cu-K α radiation ($\lambda=1.5406 \text{ \AA}$) operating at 40kV and 40mA to analyze the phases of the synthesized samples. The patterns were analyzed with DIFFRAC.EVA software and phase matching with ICDD-PDF 2011 database. A batch of samples were sintered at 300, 350, 400 and 500 °C

for three hours in order to analyze the thermal treatment effect on the glow curves and their TL response. Different annealing (100, 200, 300, 350 or 400 °C), as pre-irradiation treatment of the samples, were carried out during 30 minutes and then quenched in air by placing the samples on a cooper block at RT. All irradiations were performed in a Gammacell-3000 Elan irradiator with ^{137}Cs sources that provided 9.2 Gy min^{-1} dose rate. TL glow curves were obtained in a Harshaw TLD 3500 reader with nitrogen flow and at 2°C s^{-1} heating rate. Because the TL response depends on the mass of the sample, the mass were kept constant at 8 mg during all experiments.

3. RESULTS AND DISCUSSION

3.1 XRD of the synthesized samples

The XRD pattern of undoped sample is shown in Figure 1(a), where it is possible to assign all reflections to the LiF and NaF separated phases. The 2θ values of the experimental peaks match well with the powder diffraction files (PDF) 00-004-0857 and 01-073-1922 corresponding to LiF and NaF phases, respectively. The reflections at around 35° and 42° belong to the $K\beta$ signal of the most intense reflections of lithium fluoride and sodium fluoride phases. On the other hand, in the diffraction pattern of the Eu-doped sample (Figure 1(b)) it is possible to observe the peaks due to LiF and NaF phases, but also the presence of additional reflections situated below 30° , 42.6° and 52.5° which had been assigned to a $\text{Na}_{1.5}\text{Eu}_{1.5}\text{F}_6$ extra phase.

3.2 Glow curves of unsintered samples

The as prepared samples were placed in aluminum disks and their TL backgrounds were readout. The glow curves of samples (irradiated at 30 Gy) with different impurity ions are shown in Figure 2. The glow curves are composed by a small shoulder below 100°C , a main peak and depending on the sample, another shoulder on the right side of the principal peak appears.

When cerium or lutetium is present on the LiF+NaF lattice, the structure of the glow curves becomes complex, i.e., broad glow peaks are present. The LiF+NaF samples doped with lutetium or thallium show an intense glow peak at about 175°C and 135°C , respectively, whereas the undoped sample shows two overlapped peaks at 140°C and 165°C . The maximum TL intensity was shown by the thallium-doped LiF+NaF while the undoped sample showed the smaller one, but with simpler glow curve. The TL behavior of the samples as a function of the gamma dose was tested between 1 and 1500 Gy. The best linear dose-response intervals were shown by the undoped and the thallium-doped samples, from 10

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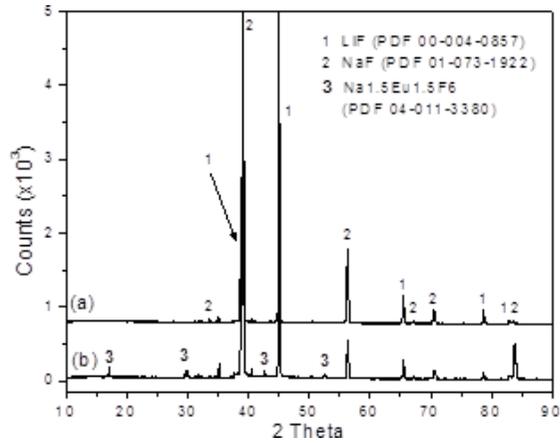


Figure 1: X-ray diffraction pattern of (a) LiF+NaF undoped sample, and (b) LiF+NaF:Eu.

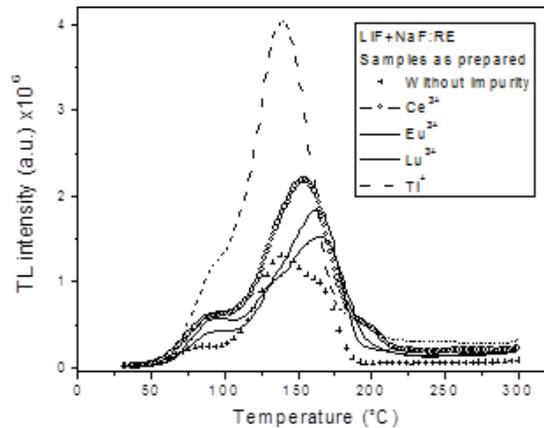


Figure 2: Glow curves of LiF+NaF samples as prepared (unsintered), pure and doped samples, with different ion impurities. All samples were irradiated at 30 Gy gamma dose from ¹³⁷Cs of Gammacell-3000 Elan-Best Theratronics.

to 500 Gy and 10 to 100 Gy, respectively. The dose-response glow curves of the undoped sample are shown in Figure 3(a), where no additional peaks above 300°C were observed. For the cerium, europium and lutetium samples, such intervals were found between 10 to 50 Gy. As is shown on Figure 3(b) the linearity loss on the latter samples was observed due to the non-equal growth of the main peak and the shoulder on the right side of the glow curve additionally to the emergence of another peak at around 375°C with higher doses.

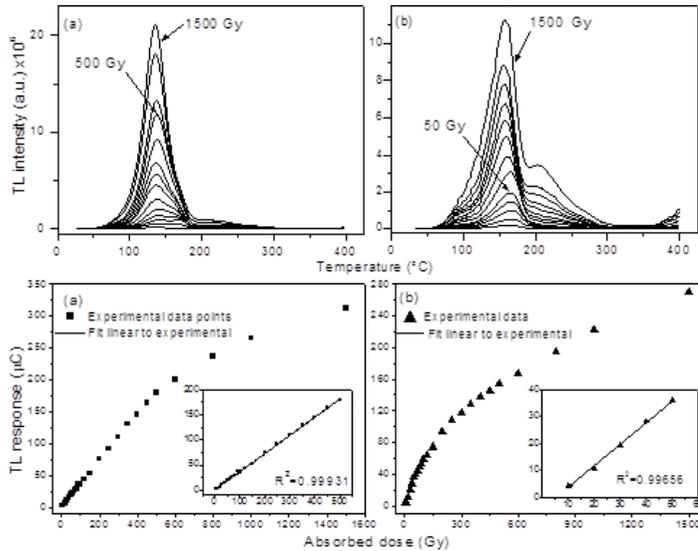


Figure 3: Glow curves (top to bottom, 1500-1 Gy), and dose-response of the unsintered samples, (a) undoped LiF-NaF, (b) Eu-doped sample. In the inset is the linear TL response stage.

In order to evaluate the behavior of the TL response to repeated irradiation-readout cycles, the samples were irradiated at 30 Gy and immediately their glow curves were obtained, this cycle was repeated several times until the TL response was stable. In Figure 4(a) are shown the glow curves of the undoped sample as an example of the observed behavior of all samples, where it was seen that the TL response increases in almost 100% of the initial response on the first seven cycles and then remains constant. This fact indicates the need of a thermal stabilization of the defects involved on the TL emission. Such stabilization was tried by providing a pre-irradiation heat (i.e. annealing treatment) during 30 minutes at certain temperature in the interval of 100°C to 400°C (Figure 4(b)). It can be observed that TL response is insensitive to the annealing treatment in the interval of 0 to 300°C, also a strong decrease on the TL intensity was observed in the thallium and cerium doped samples at an annealing temperature of 400°C. Such loss on luminescence intensity is in agreement with previous works of other authors [5, 7], where in LiF systems a strong loss on TL intensity was observed with annealing treatments above 280°C; the variance on quenching temperature on LiF-NaF system may be due to the presence of sodium on the lattice thus showing the strong effect of lithium fluoride on the system.

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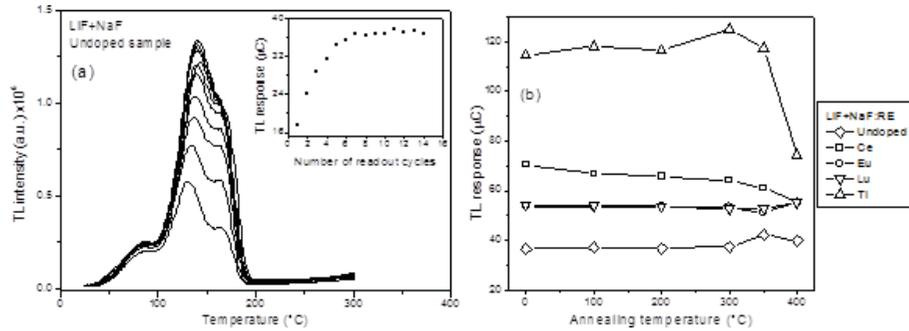


Figure 4: (a) Glow curves of continuous irradiation-readout cycles of unsintered and undoped samples, (b) TL response as a function of annealing temperature of the unsintered samples.

3.3 Glow curves of sintered samples

Different sintering treatments (300-500°C) were performed in order to evaluate its effects on the glow curve of each sample. The glow curves of the sintered samples without an annealing treatment were obtained (Figure 5) and several effects were observed: the TL intensity of the main peak changed depending on the sintering temperature; for the undoped, europium and lutetium samples such intensities were maximum with the 400°C sintering, whereas for the

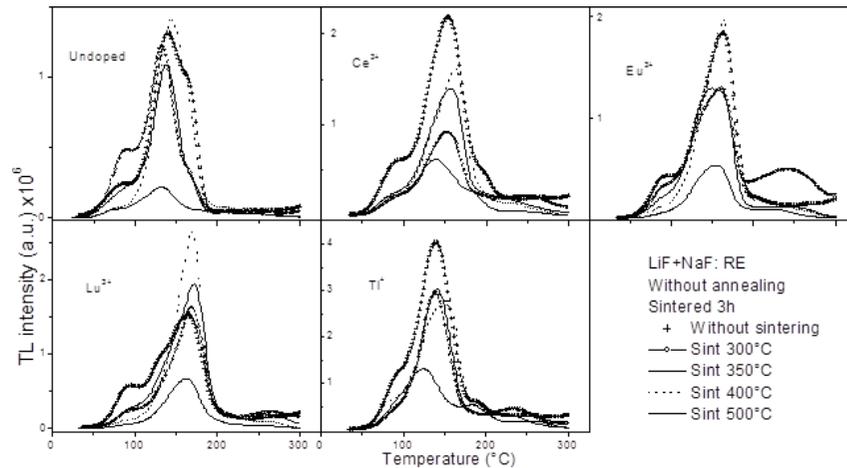


Figure 5: Glow curves, without and with sintering treatments, of undoped and RE-doped LiF+NaF samples. The irradiation dose was 30 Gy and not annealing pre-irradiation was given and the first glow peak below to 100°C was intense for RE-doped samples.

cerium and thallium doped samples this happened without sintering treatment. It was also observed that the lower sintering temperatures promote the formation of the low temperature peak below 100°C. The temperature of the maximum TL intensity (T_{Imax}) was affected as follows: for the europium and lutetium samples it remains practically constant with sintering temperatures between 0 and 400°C, whereas the other samples have small variations from 0 to 300°C showing higher T_{Imax} with a 400°C sintering. In all cases the sintering treatment at 500°C moves down T_{Imax} about 15°C regarding the higher.

Considering the glow curve structure and the intensity of the main peak, the best sintering treatments were 350°C and 400°C; the former for the undoped, europium and lutetium samples and the latter for the cerium and thallium-doped samples. In all cases the sintering at 500°C results the one with lower TL intensity and the glow curves become more complicated, i.e. an overlap and complex peak distribution is observed as well as a shift of T_{Imax} to lower temperatures.

Additionally to the sintering treatment, the effect of the annealing temperature on the TL response was studied. A heating treatment of 30 min at a certain temperature between 100 and 400°C was performed previous to the irradiations and the glow curves were obtained. In Figure 6 the TL response at different annealing temperatures for each sintering treatment is shown. The TL response of all sintered samples were found to be dependent on the annealing temperature: for the sintering treatments between 300°C and 400°C such dependence is described by a curve with a maximum at a definite temperature and then falls down, while for the 500°C sintering the behavior is interrupted

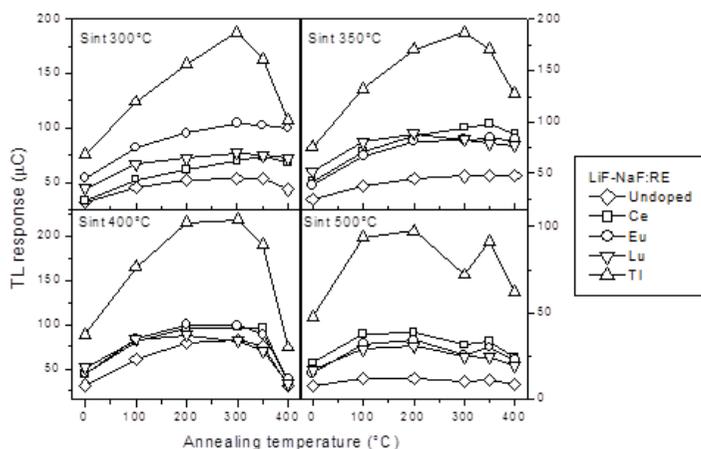


Figure 6: TL-response dependence on the annealing temperature for each sintering treatment. Annealing temperature at zero indicates the samples without annealing treatment as reference.

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by a sudden decrease of the TL intensity with an annealing of 300°C. In the case of samples sintered at 300°C and 350°C the trends in behavior and TL response values appears to be the same, however it is important to point out the differences between their glow curves: with the sintering treatment at 300°C the not well stabilized peak at around 240°C and the shoulder at the left of the main peak make a bigger contribution to the integrated TL response regarding to such peaks on the 350°C-sintered sample, that is to say, a slightly higher temperature is enough to stabilize the defects involved in the TL emission of the main peak.

The similarities on the TL behavior and structure of glow curves between the sintering treatments at 300°C, 350°C and 400°C can be attributed to the nature of the defects involved in the TL emission. Several authors [9] had reported the correlation between glow peaks and the aggregate formation of Mg^{2+} -V defects in LiF:Mg, Ti, where the main peaks 2 and 5 are due to the presence of dipoles and trimers of such defects, respectively. With LiF+NaF:RE the situation is slightly different because of the Na and RE ions presence, but it can be inferred that sintering treatments between 300°C and 400°C are enough to dissolve precipitates or aggregates of higher order but without dissolving completely the aggregates of lower order responsible of the TL emission. On the other hand, a sintering treatment at higher temperature (500°C) achieves the dissolution of even such defect aggregates, thus the TL emission is decreased and the glow curves change due to the participation of other kind of defects. A similar effect can be observed with annealing treatments; in all cases the TL emission is dropped down above certain temperature indicating the dissolution of dipoles and trimers involved in the TL emission of the main peak [2].

The best annealing conditions for each sample were chosen by taking into account the TL intensity of the main peak and the presence or absence of other peaks in the glow curve. The best thermal treatments for each sample were (sintering and annealing, respectively): 400°C and 300°C temperature for undoped samples; 350°C and 350°C for Ce-doped samples, 400°C and 300°C for Eu-doped, 400°C and 200°C for Lu-doped, and 350°C and 300°C for Tl-doped samples. Using the thermal treatments mentioned above, the thermal bleaching was performed in order to partially erase the glow curve. With this aim, the samples were bleached after irradiation during 5 minutes and the residual glow curve was obtained (Figure 7(a)). It can be observed that there are four peaks located at 156°C, 169°C, 210°C and 219°C. Performing a thermal bleaching of 150°C is possible to obtain the main peak (located at about 150°C) without the contribution of the shoulder on the left side of the glow curve and the TL intensity of the main peak remains almost the same. The thermal bleaching for Lu-doped samples is shown in Figure 7(b), where five peaks can be identified at

90°C, 170°C, 206°C, 230°C and 270°C. The best thermal bleaching was 150°C temperature although the intensity of the main peak is decreased on 30%, similar results of bleaching were observed for the other RE-doped samples.

Another purpose to make a thermal bleaching is to reduce the variation on the TL response because of the lower temperature peaks obtaining a more stable peak, i.e. at higher T_{Imax} . Thus, the TL response after several irradiation-thermal bleaching-readout cycles was evaluated for all samples. The undoped and Lu-doped samples were subjected to these cycles, in both cases it was observed a gradual decrease on the TL response because of a reduction on main peak TL intensity. These behavior could be due to more complex aggregation processes in which an accumulation of such defects after several reading cycles end up affecting the TL emission on a negative behavior [7], so more investigation is needed in that direction.

Because the thermal bleaching (Figure 7) shows several peaks are composing the complex glow curves of the LiF+NaF phosphors, a glow-curve deconvolution procedure (GCD) was applied to glow curves obtained with and without sintering treatments of pure and RE-doped LiF+NaF samples (Table 1). Five peaks were well deconvoluted from the glow curves assuming the general order kinetics [6] and the FOM were less than 1.98% which is an acceptable value. The values of the order kinetics (b) were varying from the 0.82 to 1.52 for the unsintered LiF+NaF samples. When the samples were sintered, the order kinetics were about 1 to 1.98. The activation energy (E) values were between 0.8 and 1.34 eV. The behavior of b and E parameters may ascribed to the broad complex glow curves for the unsintered LiF+NaF phosphors, and the overlapped glow peaks in the whole glow curves of the sintered LiF+NaF samples.

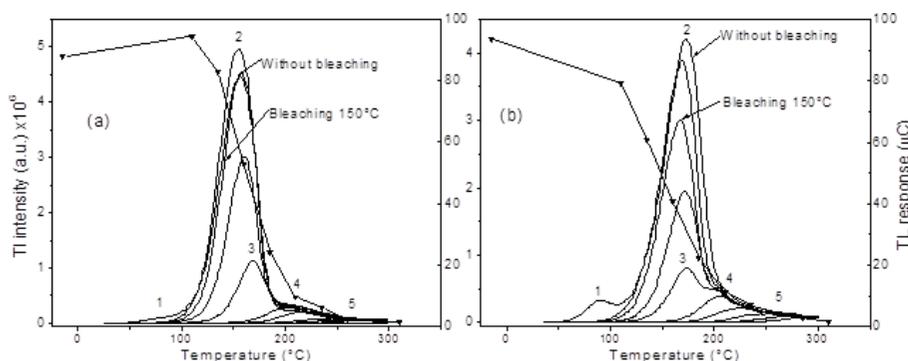


Figure 7: Thermal bleaching of (a) undoped LiF+NaF sample, and (b) Lu-doped sample.

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Table 1: Kinetics parameters of unsintered and sintered of LiF-NaF phosphor. The corresponding units are E (eV), s (s^{-1}) and T_{max} ($^{\circ}C$).

| Sample | Kinetic parameters | Peak 1 | Peak 2 | Peak 3 | Peak 4 | Peak 5 | FOM (%) | |
|--------------|--------------------|----------|----------|----------|----------|----------|---------|------|
| LiF-NaF pure | b | 1.70 | 1.32 | 1 | 2 | 1.62 | 0.82 | |
| | E | 0.791 | 1.124 | 1.159 | 1.131 | 1.138 | | |
| | T_{max} | 79.63 | 136.6 | 162.8 | 204 | 249 | | |
| | s | 7.16E+10 | 2.61E+13 | 9.03E+12 | 2.34E+10 | 2.28E+10 | | |
| | b | 1.24 | 1.45 | | 1.27 | 1.82 | | |
| | E | 0.8 | 1.149 | | 1.15 | 1.16 | | |
| Sintered | T_{max} | 77.32 | 149.85 | | 212.46 | 262.49 | 1.98 | |
| | s | 1.20E+11 | 1.83E+13 | | 2.41E+11 | 1.82E+10 | | |
| | b | 1.34 | 1.08 | 1 | 1.07 | 2 | | 1.52 |
| | E | 0.8 | 0.81 | 1.2 | 1.3 | 1.34 | | |
| | T_{max} | 86.83 | 151.57 | 194.46 | 221.01 | 257.72 | | |
| | s | 5.60E+10 | 1.07E+09 | 2.76E+12 | 5.63E+12 | 1.37E+12 | | |
| b | 1.29 | 1 | 1.78 | 1.52 | 2 | | | |
| E | 0.82 | 0.85 | 1.23 | 1.32 | 1.34 | | | |
| Sintered | T_{max} | 89.07 | 156.71 | 194.60 | 222.80 | 254.80 | 1.66 | |
| | s | 9.21E+10 | 2.96E+09 | 5.61E+12 | 7.89E+12 | 1.63E+12 | | |
| | b | 1.54 | 1.62 | 1.01 | 1.50 | 2 | | 0.88 |
| | E | 0.94 | 1.01 | 1.05 | 1.12 | 1.1 | | |
| | T_{max} | 87.54 | 135.51 | 161.99 | 198.78 | 257.17 | | |
| | s | 5.59E+12 | 9.72E+11 | 4.71E+11 | 2.60E+11 | 6E+09 | | |
| b | 2 | 1.07504 | | 1.98701 | 2 | | | |
| E | 0.94 | 1.05 | | 1.12 | 1.13 | | | |
| Sintered | T_{max} | 81.43 | 157.61 | | 199.75 | 237.67 | 1.19 | |
| | s | 9.48E+12 | 6.36E+11 | | 2.36E+11 | 2.61E+10 | | |
| | b | 1.21 | 2 | 1.10 | 1.30 | 2 | | 0.98 |
| | E | 0.9 | 0.94 | 1.03 | 1.12 | 1.34 | | |
| | T_{max} | 89.29 | 131.59 | 167.30 | 205.04 | 263.92 | | |
| | s | 1.30E+12 | 1.59E+11 | 2.31E+11 | 1.79E+11 | 1.00E+12 | | |
| b | 2 | 1.17463 | | 1.04328 | 2 | | | |
| E | 0.92 | 1.05 | | 1.12 | 1.34 | | | |
| Sintered | T_{max} | 87.02 | 164.69 | | 209.89 | 249.78 | 1.13 | |
| | s | 2.92E+12 | 3.87E+11 | | 1.36E+11 | 2.21E+12 | | |
| | b | 2 | 1.41 | 1.62 | 1.9 | 2 | | 1.35 |
| | E | 0.92 | 1.01 | 1.29 | 1.32 | 1.33 | | |
| | T_{max} | 94.12 | 142.09 | 193.75 | 232.44 | 266.52 | | |
| | s | 1.89E+12 | 6.00E+11 | 3.63E+13 | 2.54E+12 | 7.95E+11 | | |
| b | 1.82 | 1.09 | 1 | 2 | 1.73 | | | |
| E | 0.95 | 0.97 | 1.28 | 1.3 | 1.33 | | | |
| Sintered | T_{max} | 92.93 | 145.79 | 180.55 | 204.97 | 235.84 | 1.06 | |
| | s | 4.74E+12 | 1.77E+11 | 6.14E+13 | 1.58E+13 | 4.24E+12 | | |

4. CONCLUSIONS

The undoped and rare earth-doped LiF+NaF phosphor powder was synthesized by solid state reaction, both phases for all samples were identified by the XRD analysis. Samples as synthesized, i.e., without

sintering treatment was found insensitive to pre-irradiation annealing, while the sintering samples (300, 350, 400 or 500 °C) the thermoluminescent (TL) response was dependent on the annealing temperature (100-400 °C). The thermal effect of the annealing on the sintered samples gives enough sensitivity to gamma radiation of ^{137}Cs obtaining good TL response. The glow peak located below 100°C was erased with thermal bleaching at 150°C and it improved the TL signal reproducibility up to four times. The main glow peak for all rare earth (Ce^{3+} , Eu^{3+} , Lu^{3+} or Tl^+ , at 0.5 mol%) in the LiF+NaF samples was located at about 150°C, that may be useful as dosimetric peak for dosimetry purpose. The glow curves of the LiF+NaF phosphor were well deconvoluted by five peaks assuming the general order kinetics. The variation of the values of the kinetics order (b) and the activation energy (E) parameters may related to the broad complex glow curves for the unsintered LiF+NaF phosphors, and the overlapped glow peaks present in the glow curves. Finally, from the observed glow curves and TL characteristics of the LiF+NaF:RE phosphors make attractive to be useful in dosimetry at low doses (<50Gy) and as detector dosimeter. Further work is needed to identify the nature of the defects involved in the sintering samples, which modify the intensity and the shape of the glow curves.

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