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Thermoluminescence dosimetry properties and kinetic analysis of $MgSO_4$:Dy microcrystalline prepared by solid state method

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HIGHLIGHTS

- $MgSO_4$:Dy was produced for the first time by melting method.
- Its TL sensitivity is 25 times higher than LiF:Mg,Ti.
- Kinetic parameters were obtained using different methods.
- TL dose response is linear up to 10 Gy.
- It is recommended as high sensitive TL dosimeter.

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ABSTRACT

$MgSO_4$:Dy phosphor was synthesized by solid state method. Sample characterization was carried out using X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS). Highest thermoluminescence (TL) sensitivity was observed at 0.3 mol% of dysprosium impurity. T_m-T_{Stop} method was used to identify the number of glow peaks enclosed in TL glow curve. Computerized glow curve deconvolution (CGCD), initial rise and variable heating rate methods were used to determine kinetic parameters. TL sensitivity of produced $MgSO_4$:Dy phosphor is approximately 8.5 times more than LiF:Mg,Ti (TLD-100). Other dosimetry features of the synthesized phosphor are presented and discussed.
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1. Introduction

Sulfates doped with different impurities are known as proper TL materials. Different types of sulfates have been synthesized to examine their TL characteristics. $CaSO_4$, $MgSO_4$ and $BaSO_4$ are the most familiar kinds of these materials.

$CaSO_4$:Dy was known as a suitable dosimeter because of its high TL sensitivity and simple preparation methods. It has been employed extensively for environmental radiation measurements and personal monitoring (Yamashita et al., 1968, 1971). Also it has been found that $CaSO_4$:Dy with high temperature TL peaks can be used in reactor or hot waste dosimetry (Mathur et al., 1999).

Along with calcium sulfate, other types of sulfates like $SrSO_4$ and $BaSO_4$ (Dixon and Ekstrand, 1974) and also $MgSO_4$ have been studied. It has been found that pure magnesium sulfates does not show good TL properties (Luo et al., 1999a). So, this material was synthesized with different impurities (Luo et al., 2006; Manam and Das, 2010; Zhang et al., 2002). Dysprosium due to its desirable properties was used as dopant more and more (Upadhyay et al., 2011). Zhang et al. (2000) synthesized $MgSO_4$:Dy (0.1 mol%) with hot sulfuric acid. TL glow curve of irradiated $MgSO_4$:Dy was deconvoluted into four glow peaks at about 135, 196, 262 and 361 °C based on Gaussian function fitting. In another investigation, Zhang et al. (2001) synthesized $MgSO_4$:Dy with above mentioned method. TL spectra of this material following 10 kGy gamma irradiation had four glow peaks at ~140, ~190, ~260, ~360 °C.

Kher et al. (2008) also studied the luminescence behavior of $MgSO_4$:Dy. First this substance was synthesized by solid state diffusion method and then TL glow curve of gamma irradiated

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$MgSO_4$:Dy was recorded and two peaks at ~110, ~220 °C were identified. The amount of dysprosium was optimized at 0.1 mol% for this phosphor.

$MgSO_4$:Dy nano-rods was also synthesized and TL glow curve of this material following gamma irradiation was deconvouluted by using general order (GO) model. The main glow curve was decomposed into four peak components at 416, 436, 489, 550K. Dose response of $MgSO_4$:Dy nano-rods was linear up to absorbed dose of 10^4 Gy (Zahedifar et al., 2016).

In this work, $MgSO_4$:Dy was synthesized by solid state method. This technique is much safer than hot sulfuric acid method and TL sensitivity of the produced phosphor is much higher than those of all pre-mentioned methods.

2. Experimental

The precursors used in this work are $Mg(NO_3)_2 \cdot 6H_2O$(99.99% purity), $(NH_4)_2SO_4$(99.99% purity) and $Dy(NO_3)_3$, all prepared from Merk Chemicals. For synthesizing $MgSO_4$:Dy, 0.8 g of $Mg(NO_3)_2.6H_2O$(99.99% purity), 0.4 g of $(NH_4)_2SO_4$(99.99% purity) and 0.007 g (0.5 mol%) of $Dy(NO_3)_3$ were rubbed in a mortar and mixed together very well. Then the mixture was put in a crucible and placed in a furnace at 600 °C for 50 min. Then the produced white powder was rubbed until a uniform white powder of $MgSO_4$:Dy was obtained.

Structure of the synthesized $MgSO_4$ was confirmed by X-ray diffraction (XRD) pattern using a Rigaku D-max III diffractometer (Bruker D8 Advance) with CuK_α ($\lambda = 1.54°$ A) radiation under the conditions of 40 kV and 30 mA, at a step size of $2\theta = 0.02°$. The shape of the produced $MgSO_4$ phosphor and elemental concentrations were recognized by a scanning electron microscope model Philips XL-30 ESEM equipped with energy dispersive spectrometer (EDS). The mass of samples was fixed at 0.004 ± 0.0001 g during all the experiments by using a SHIMADZU Ax120 (max = 120 g, d = 0.1 mg). Irradiations were performed using a ^{60}Co gamma source. The TL readouts were carried out in a Harshaw model 4500 computer-based TL reader using a contact heating with a precision of 1 °C. The temperature of the heater strip was recorded as indicator of temperature of the sample. The heating rate for readout was 2 °C/s (with preheat of 50 °C) to a maximum temperature of 350 °C. All the samples were heated and annealed in a programmable oven with temperature precision of 1 °C and then were cooled rapidly to room temperature (75 °C/min).

3. Results and discussion

Impurities are of crucial importance in creating trapping centers or acting as activators in the host material. Therefore a proper dopant with optimum concentration can fundamentally improve the TL sensitivity. Here, dysprosium was chosen as dopant. $MgSO_4$ phosphor was produced with different amount of this impurity and the TL responses were recorded for the same conditions of annealing, reading conditions and the same absorbed dose of 500 mGy. Shown in Fig. 1 (a) are the TL responses (the area underneath the glow curve) of $MgSO_4$:Dy for different amounts of Dy which indicates that the most sensitivity yields for 0.3 mol% of impurity. For each impurity concentration, the sample preparation was repeated for three times. The average TL responses along with uncertainties for different impurity concentrations are shown in Fig. 1(a). For better comparison, TL glow curves of $MgSO_4$:Dy phosphor for different amount of dysprosium dopant after 500 mGy gamma irradiation are shown in Fig. 1(b). This figure also reveals that the optimum concentration is achieved at 0.3 mol% of Dy.

Presented in Fig. 2 is the XRD pattern of $MgSO_4$ microcrystalline confirming an orthorhombic structure that is in correspondence with ICSD collection code no 74–1364. The shape and size of the synthesized $MgSO_4$ powder particles are shown in SEM image of Fig. 3. Since the XRD pattern cannot reveal the presence of impurity with low concentration, EDS analysis was carried out to estimate the elemental concentration in produced $MgSO_4$:Dy phosphor. The result is shown in Fig. 4 and the percentages of elements are shown in the inset of the figure. EDS analysis was used to investigate the chemical composition and purity of products. The EDS spectrum of $MgSO_4$:Dy sample shows characteristic signals of Mg, S, O and Dy (as dopant) elements as is shown in Fig. 4. In this figure, there is no trace signals related to other elements, as indication of proper sample. The weight percent of elements detected by EDS analysis and calculated are: Mg (EDS:15%, cal:20%), S (EDS:34.95%, cal:27%), O (EDS:44.82%, cal:53%). EDS is most often used for qualitative elemental analysis, in other words it is used to simply determine which elements are present in the sample and what is their relative abundance, furthermore only the main K_α lines of Mg, S and O are present in EDS spectrum. So differences between theoretical and calculated values is due to EDS limitations.

Fig. 5 shows TL glow curves of $MgSO_4$:Dy phosphor and LiF:Mg,Ti (TLD-100) after 500 mGy gamma ray irradiation. Inserted in top right of Fig. 5 is a comparison between TL sensitivities of LiF:Mg,Ti (TLD-100) and $MgSO_4$:Dy phosphor synthesized by hot sulfuric acid (Luo et al., 1999b). which exhibits that TL intensity of $MgSO_4$:Dy phosphor is about 8 times less than TLD-100. $MgSO_4$:Dy phosphor produced in this work is about 8.5 times more sensitive than TLD-100. This is the highest TL sensitivity observed in $MgSO_4$ phosphor . As mentioned above, all samples had the same weight and comparison of TL response was carried out considering corrections due to different masses.

To study TL properties of a certain material, it is necessary to find a thermal regime that optimizes the TL response. Proper annealing regime, in addition to empty the traps remained occupied in preceding irradiations, causes restoration of centers or clusters responsible for a trapping state which in turn improves TL sensitivity of the material. An annealing program of 900°C-1h has been recommended for $MgSO_4$:Dy powder prepared by hot sulfuric acid (Zhang et al., 2001). For finding the best annealing program, different temperatures between 400 and 900 °C with same duration of 30min were tested. Afterwards, different anneal periods were also tested. As is shown in Fig. 6, the most proper annealing regime was achieved at 700°C-30 min.

Fig. 7 shows the dose response of $MgSO_4$:Dy phosphor in a log-log scale. In a log-log scale, a straight line with slop 1 corresponds to linear dose response. It is seen that the dose response is linear up to absorbed dose of 10Gy and then becomes sublinear. Saturation at low amounts of absorbed dose is a common behavior of high sensitive TL materials. In another work on TL dose response of $MgSO_4$:Dy phosphor following gamma irradiation, a linear dose response up to absorbed dose of 930Gy was reported and in higher doses saturation was occurred (Upadhyay et al., 2011). TL dose response for $MgSO_4$:Dy nano-rods with very low TL sensitivity is linear until the absorbed dose reaches 10^4Gy (Zahedifar et al., 2016).

For testing the reusability of $MgSO_4$:Dy material, seven sample of magnesium sulfate with the same weight were prepared, then they were subjected to annealing, 10Gy irradiation followed by readout and these cycles were repeated for 10 times. The results is depicted in Fig. 8. As is observed, within a low uncertainty, the TL response remains unchanged upon 10 above cycles.

TL signal stored in a phosphor by irradiation with ionizing radiation fades with storing the sample at room temperature. This phenomenon is more significant for low temperature peaks and depends upon kinetic parameters such as activation energy and

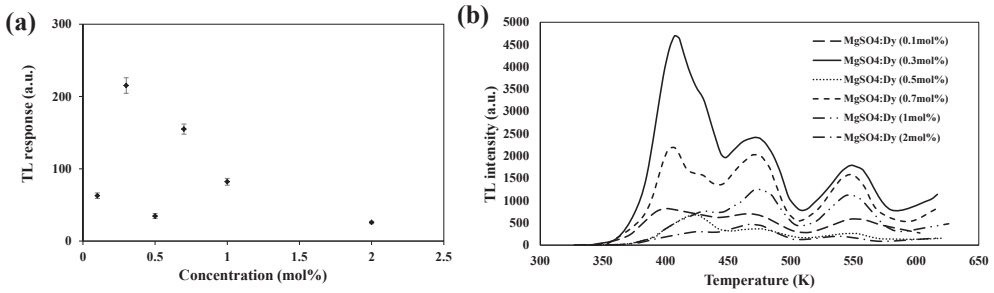

Fig. 1. (a) TL response for different impurity concentrations in $MgSO_4$:Dy phosphor. (b) TL glow curves of $MgSO_4$:Dy for different concentration of dysprosium after 5Gy gamma irradiation.

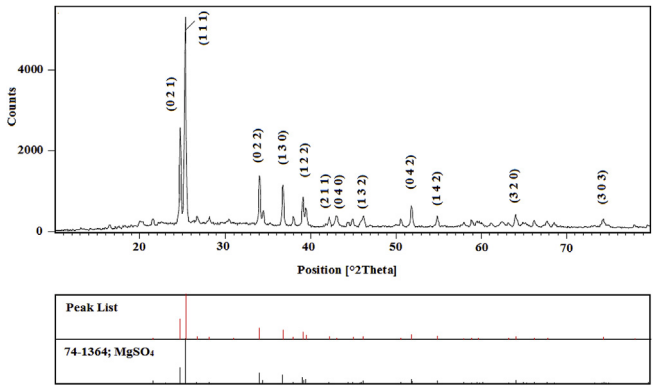

Fig. 2. XRD pattern of $MgSO_4$ phosphor which reveals the orthorhombic structure without additional phases.

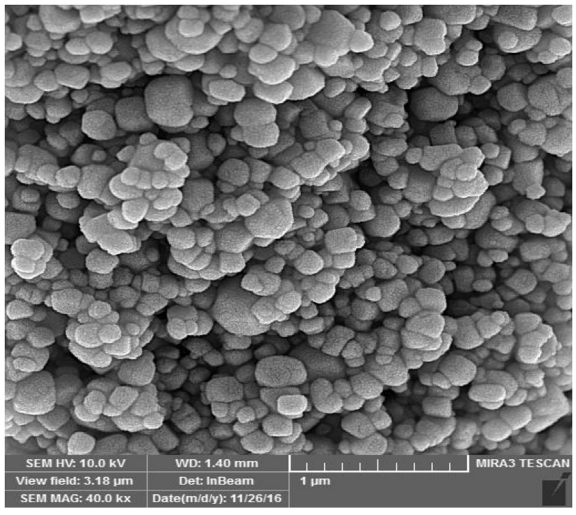

Fig. 3. SEM image of the produced spherical shaped $MgSO_4$ microcrystalline.

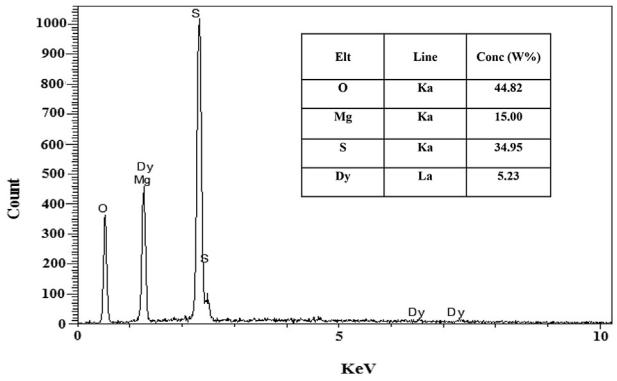

Fig. 4. EDS pattern of $MgSO_4$:Dy phosphor which confirms the formation of the desired phosphor without contamination.

frequency factor. For dosimetry purposes, it is required that the stored TL signals to be stable during irradiation and readout the sample, so fading is an undesirable property. The plots that show fading of $MgSO_4$:Dy microcrystalline are depicted in Fig. 9 (a, b). In part (a) it is obvious that the intensity of first peak at about 407 K is declined around 19.7% after one month, while intensities of second and third peaks remain almost unchanged after 20days storage at room temperature. Shown in Fig. 9 (b) are the glow curves immediately after irradiation, one day and 20 days after irradiation.

Deconvolution of TL glow curve was carried out by means of CGCD program produced in our laboratory using Levenbery-Marquart algorithm. This program is capable of fitting experimental glow curves to those generated by deconvolution functions describing general order of kinetics, mixed order of kinetics and also functions describing continuous trap distribution to obtain kinetic parameters of glow peaks. For the TL glow curves having kinetic orders close to 1or 2, respectively the first and second order kinetics models are sufficient to determine the kinetic parameters, but for the kinetic orders significantly far from the limiting values 1and 2, it is essential to use general order kinetics. In this work, it can be observed that b values determined by isothermal decay method for each peak are between 1 and 2 and not close to 1 or 2, so GO model was used for deconvolution procedure.

Solution of rate equations in TL process governing transport of charge carriers between trapping states, conduction band and recombination centers in general order kinetics in terms of intensity and temperature of the peak maximum is given by (Kitis et al., 1998):

$$I(T)=I_m b^{\frac{b}{b-1}}\exp\left(\frac{E(T-T_m)}{kTT_m}\right)\times\left\{\frac{T^2}{T_m^2}(b-1)\left(1-\frac{2kT}{E}\right)\exp\left(\frac{E(T-T_m)}{kTT_m}\right)+1+(b-1)\frac{2kT_m}{E}\right\}^{\frac{-b}{b-1}} \quad (1)$$

In this equation, I_m, T_m(K), T(K), E (eV), k (eV/K), b; are TL intensity, maximum temperature, absolute temperature, activation energy, Boltzmann's constant and kinetic order correspondingly.

Usually, the precision of fitting is examined by the figure of merit (FOM) (Balian and Eddy, 1977):

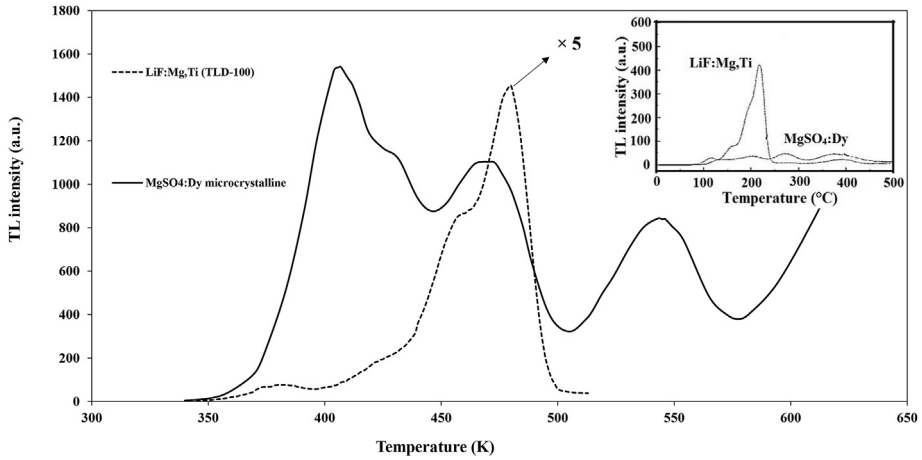

Fig. 5. Comparison of TL sensitivities of $MgSO_4$:Dy powder and LiF:Mg,Ti (TLD-100) after 500 mGy gamma irradiation. Shown at top right of figure are TL glow curves of $MgSO_4$:Dy (Luo et al., 1999b) and LiF:Mg,Ti.

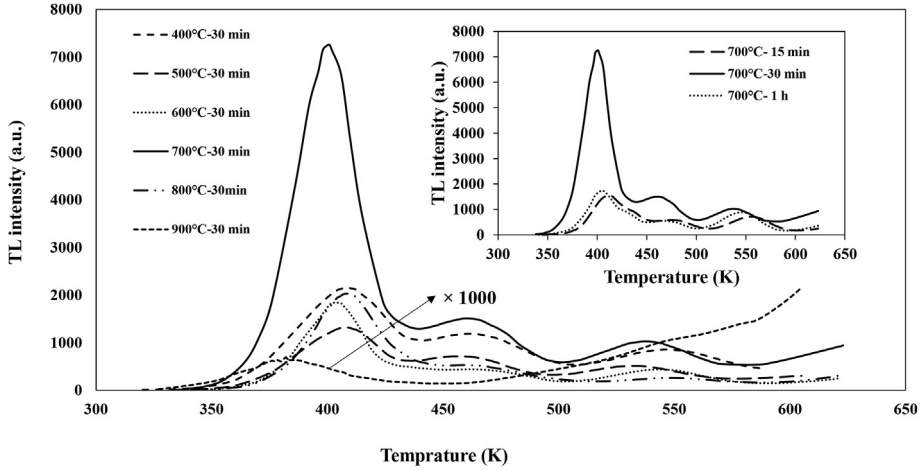

Fig. 6. TL glow curves following different annealing regimes from 400 °C up to 900 °C and duration of 30 min which reveals the 700 °C is the optimum temperature. The inset figure at top right assures that the annealing time of 30 min is the optimum annealing time.

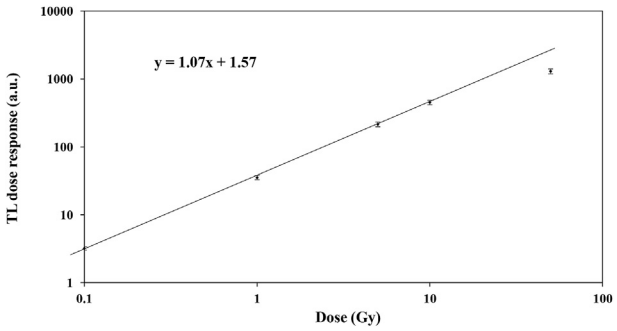

Fig. 7. TL dose response of $MgSO_4$:Dy phosphor.

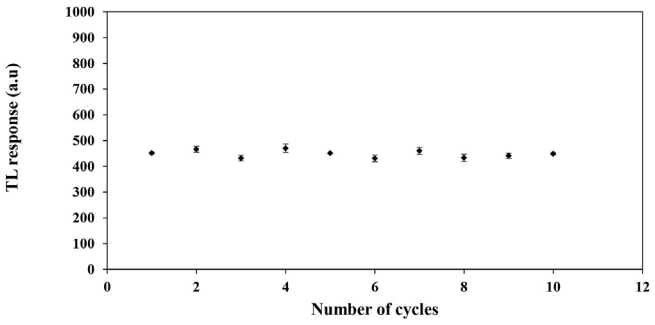

Fig. 8. Reusability of $MgSO_4$:Dy phosphor after 10Gy gamma irradiation.

$$FOM = \sum_{jf}^{jl} \frac{100[Yi - Y(Xi)]}{A} \quad (2)$$

where j_f and j_l are the first and last temperature interval ΔT that used for curve fitting, Y_i and $Y(X_i)$ are both the intensity of the ith interval, the first is obtained from experiment and the second is expected from Eq (1). FOM values lower than 2.5% mean that a good harmony exists between theoretical and experimental results.

$T_m - T_{stop}$ is an efficient method to identify the number of glow peaks accommodated in the main glow curve. In this method, firstly the irradiated sample (here the samples were irradiated to 5 Gy dose from ^{60}Co) is heated in TL reader with a constant heating rate (here 5 °C/s) to a certain temperature known as T_{stop}, then is cooled to room temperature in TL reader without removing the sample

and is heated again with a constant heating rate (here 2 °C/s) for recording the TL glow curve. The maximum temperature in TL glow curve is recorded as T_m. This procedure was repeated in this work for increased T_{stop} in temperature interval between 90 and 350 °C with steps of 10 °C for T_{stop} and different T_m values related to each T_{stop} were obtained. The plot of T_m against T_{stop} is presented in Fig. 10 for $MgSO_4$:Dy phosphor. As is seen in this Figure, by raising T_{stop}, the peak temperature T_m is also increased, however this increment is not smooth and several jumps are observed. In T_m-T_{stop} plot, each single glow peak is characterized by a plateau region. For glow peak which behaves as near first-order kinetics the plateau with lower slop and for the one with higher kinetic order, the plateau with higher slop is expected (McKeever, 1985).

After determination of the number of glow peaks by T_m-T_{Stop} method and the kinetic orders from isothermal decay curve (discussed below), these parameters along with T_m and I_m estimated from the glow curve, were used as input parameters for CGCD in order to get a rapid convergence of the fitting program and obtain more accurate values for the kinetic parameters. Results are shown in Fig. 11. It can be seen that TL glow curve of $MgSO_4$:Dy includes four glow peaks at 400, 426, 461, 539K. The kinetic parameters extracted from computer fitting are shown in Table 1. As mentioned before, in previously reported work on $MgSO_4$:Dy phosphor, the TL glow curve was deconvoluted in four peaks at 135, 196, 262, and 361 °C by using Gaussian function for deconvolution process (Zhang et al., 2000). Although the number of component glow peaks obtained in this work is the same as the above report, but it is evident that general order glow curve deconvolution function applied in this work results in more accurate values for the kinetic parameters.

In order to validate the TL kinetic parameters of this phosphor, other methods of kinetic analysis including variable heating rate, isothermal decay and initial rise were also used moreover the CGCD. Isothermal decay method was used to obtain the order of kinetics, b (Chen, 1983). Solving the intensity equation in general order case for a constant temperature yields:

$$\log\left(\frac{dI}{dt}\right) = \log\left(\frac{c}{1/b-1}\right) + \left(2-\frac{1}{b}\right)\log(I) \qquad (3)$$

Where I is the TL intensity, $\frac{dI}{dt}$ the slop of isothermal decay plot for a given time, b the kinetic order and c, a constant. A plot of log ($\frac{dI}{dt}$) versus log(I) gives a straight line with the slope of (2−1/b). Except for the small satellite peak 2 for which it was not possible to record a precise isothermal decay plot and its kinetic parameters was estimated only through CGCD method, the order of kinetics for other glow peaks were evaluated by this method. For recording isothermal decay curves the following criteria should be satisfied: at first, all the lower temperature peaks should be removed by thermal bleaching and the constant temperature of decay should be selected in a temperature interval such that the higher temperature peak do not contribute in isothermal decay plot. Fig. 12 shows the plot of log($\frac{dI}{dt}$) versus log(I) for first, third and fourth glow peaks from which b was calculated by the slops of three obtained straight lines.

The TL intensity in general order of kinetics for peak maximum is:

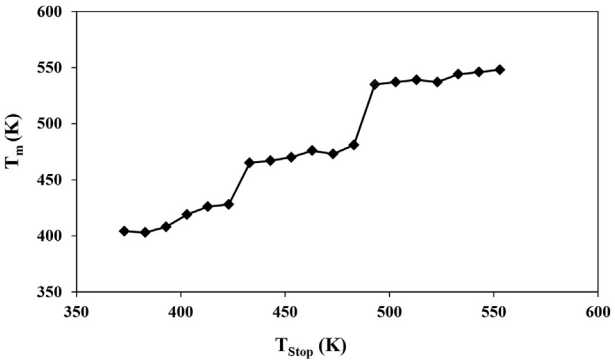

Fig. 10. T_m-T_{stop} plot of $MgSO_4$:Dy phosphor. Each plateau region indicates that a single glow peak exists in that region.

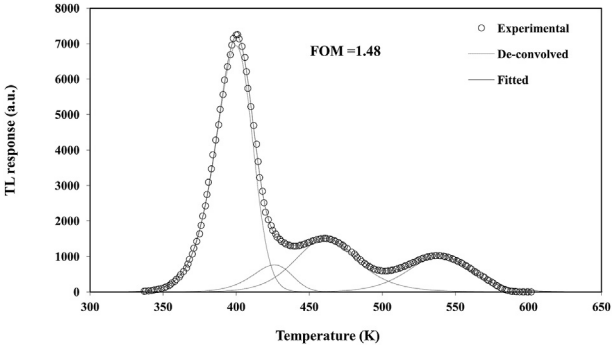

Fig. 11. TL glow curve of $MgSO_4$:Dy phosphor along with the component glow peaks obtained from CGCD.

Table 1
Kinetics parameters of $MgSO_4$:Dy phosphor obtained by CGCD method.

Im (a.u.)	Tm (K)	E (eV)	b	Peak number
12484	400	1.22	1.27	1
1373	426	1.25	1.20	2
2688	461	1.18	1.93	3
1821	539	1.51	1.67	4

The TL intensity in general order of kinetics for peak maximum is:

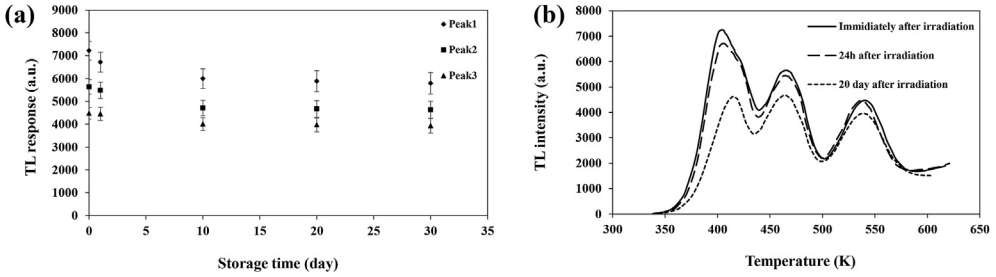

Fig. 9. (a) Fading of TL signal with the storage time for three main peaks of $MgSO_4$:Dy after one month. (b) TL glow curves of $MgSO_4$:Dy phosphor for different storage times.

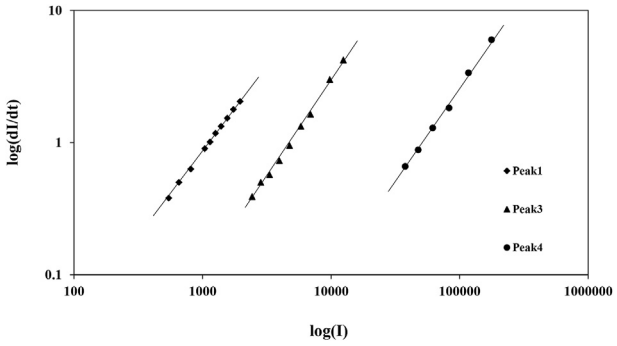

Fig. 12. Plot of $\log\left(\frac{dI}{dt}\right)$ versus $\log(I)$ for glow peaks 1, 3 and 4. The slope of each line is $\left(2-\frac{1}{b}\right)$ from which the kinetic order can be evaluated.

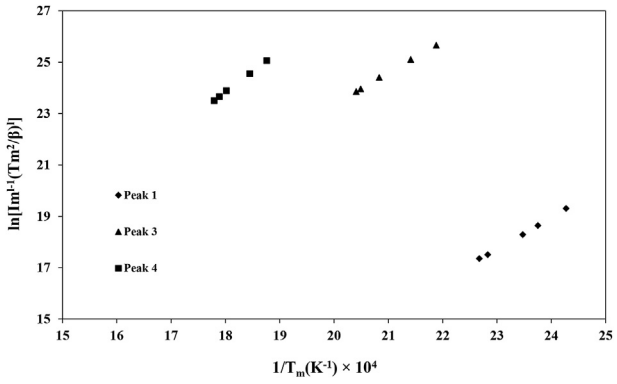

Fig. 13. $[I_{\downarrow}m^{\uparrow}(b-1)[([T_{\downarrow}m]^{\uparrow}2/\beta)]^{\uparrow}b$ against $\frac{1}{T_m}$ for different glow peaks of $MgSO_4$:Dy phosphor. Each straight line was obtained using heating rates from 1 to 5 °C/s, the slope of each line is $\frac{E}{K}$.

Table 2
Kinetics parameters of $MgSO_4$:Dy phosphor evaluated from variable heating rate, isothermal decay and initial rise methods.

Peak	b (Isothermal decay)	EVH (eV) (Various heating rate)	EIR (eV) (Initial rise)	s (s−1)
1	1.45	1.08	1.18	1.16 × 1012
2			1.13	9.72 × 1013
3	1.85	1.08	1.2	4.84 × 1010
4	1.78	1.37	1.3	5.62 × 1011

$$I_m^{b-1}\left([T_{\downarrow}m/\beta]^{\uparrow}2\right)^b = (sn_0)^{-1}((n_{\downarrow}0E)/bk)^b \exp\left(\frac{E}{KT_m}\right) \quad (4)$$

Where T_m is the peak temperature, β the heating rate, E the activation energy, k the Boltzman's constant and s the frequency factor and n_0, the initial concentration of trapping state. Plot of ln $[I_{\downarrow}m^{\uparrow}(b-1)[([T_{\downarrow}m]^{\uparrow}2/\beta)]^{\uparrow}b]$ versus $\frac{1}{T_m}$ for different heating rates gives a straight line with slope of $\frac{E}{K}$ .Using this method, the only parameters needed for calculating the activation energy are peak maximum temperature and peak intensity. Hopefully, these parameters can be read exactly from the glow curve and the obtained activation energy is not influenced by thermal quenching. In fact, this is the benefit of various heating rate method (McKeever, 1985). For recording the variable heating rate plots, sample was heated in temperature range 50–350 °C by five different heating rates of 1–5 °C/s. Drawn in Fig. 13 are the plot of ln $[I_{\downarrow}m^{\uparrow}(b-1)[([T_{\downarrow}m]^{\uparrow}2/\beta)]^{\uparrow}b]$ against $\frac{1}{T_m}$ for different separated peaks of $MgSO_4$:Dy phosphor.

Initial rise is the other method for determination of activation energy. A linear relation between TL intensity and exp $(\frac{-E}{KT})$ can be observed in initial rise part of a TL glow peak, since in this region the concentration of charge carriers in traps can be assumed as a constant regardless of the order of kinetics. Because of this relation, plotting lnI against $\frac{1}{T}$ gives a straight line that its slop is equal to $\frac{-E}{T}$. Therefore, the activation energy can be obtained from this plot. The prementioned condition is true just for initial rise part of the glow peak and for obtaining more accurate values for the activation energy, corrections should be made due to using higher intensities (Singh et al., 1988). For obtaining lnI the highest peak intensity that used in this work was about 10% of the maximum peak intensity, so the corrections for the activation energies were estimated. The comparative results of three different methods are shown in Table 2 for $MgSO_4$:Dy. As is clear, a good harmony exists between them, indicating the reliability of the obtained results for kinetic parameters.

4. Conclusion

$MgSO_4$ with optimized concentration of 0.3 mol% dysprosium impurity was synthesized by solid state method. The prepared phosphor has high TL sensitivity compared to other commercial TL dosimeters. The area of the glow peaks 2, 3 are almost unchanged by storing at room temperature. Besides simple preparation method, it is fairly reproducible with approximately the same TL response. A challenge in determining TL kinetic parameters by CGCD method is correct estimation of the number of component glow peaks contained in the complex glow curve. It is likely that glow curves simulated by the program with different number of glow peaks coincide fairly to the experimental glow curve. Therefore, it is important to realize the number of glow peaks separately. Kinetic parameters obtained by CGCD method are reliable since the number of component glow peaks were firstly identified by Tm-Tstop method and the peak temperatures and peak maxima for different glow peaks were estimated accurately from the shape of the glow curve as the input parameters. Also, the order of kinetics except for peak 2 were evaluated by isothermal decay method and inserted in the computer program as input parameters which caused rapid convergence of the glow curve deconvolution procedure and accurate estimation of activation energies.

The precision of the kinetic parameters was guaranteed by evaluating those using different methods of variable heating rate, isothermal and initial rise. The results in Table 2 reveal that they are in accordance with each other. Regarding TL dosimetry features of the synthesized MgSO4:Dy microcrystalline, it can be used as a highly sensitive TL dosimeter.

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