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Research Article

Keywords: Synthesized Li₂B₄O₇, lithium, gamma rays

Posted Date: August 24th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-841528/v1>

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Synthesized $\text{Li}_2\text{B}_4\text{O}_7$: Mg,Cu nanoparticles: a suitable thermoluminescence dosimeter for detection of high doses of gamma rays

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Abstract:

Lithium tetraborate nanoparticles co-doped with various percentages of Cu and Mg impurities were synthesized through the combustion process. Scanning electron microscopy images along with X-Ray diffraction pattern confirmed the shape and structure of the products. The Williamson-Hall equation was used to measure the size of nanoparticles that resulted in approximately 47 nm for the crystallite size. The optical band gap of about 3.7 eV was obtained for the nanostructures from the UV-visible spectrum. Furthermore, the thermoluminescence features the samples under gamma irradiation were studied at ambient conditions. The highest thermoluminescence sensitivity achieved at 0.02 % wt Cu and 0.5 % wt Mg impurities simultaneously. The results show that the co-doped nanoparticles have a linear dose response up to an administrated dose of 30 kGy and about 10% of the thermoluminescence signal fades after 30 days of storage at room temperature.

Keywords: Combustion Method, Nanoparticle, Lithium tetraborate, Thermoluminescence, Band gap, Dosimetry.

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1. INTRODUCTION

Ideal thermoluminescence (TL) materials should possess specific features, including linear dose-response, accuracy, near tissue equivalence, high sensitivity, excellent stability, well-defined TL glow curve, and a simple annealing process. So far, many dosimetric materials are employed in radiation dosimetry, and a lot of new compounds have been synthesized, but none of them matches all the desired characteristics of an ideal detector. Therefore, attempts have always been made to either prepare new TL dosimeters (TLDs) with better properties or simply improve the already existing dosimetric materials by varying the concentration of impurities or co-doping the phosphor with other elements or doping new impurities in new structures [1]. The glow peaks in the TL glow curves are explained by special parameters such as Kinetic parameters and the number of trapped electrons [2, 3]. So far, different materials have been synthesized to study their TL response in different ionizing radiation fields [4]. Amongst different TL phosphors, lithium borates due to their tissue equivalence are of great interest in TL dosimetry. In this regard, lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$) crystal is widely used for radiation measurement, especially radiation therapy and personal dosimeters because its effective atomic number (~ 7.3) is very close to the soft tissue of the body (~ 7.4) [5]. Indeed, the lithium borate dosimeters are superior to many which is used as a TL dosimeter in terms of tissue equivalence [5-9]. Besides, most of the borates are relatively stable chemical compounds and easy to handle. Furthermore, by adding elements as activators, the resultant materials will have high sensitivity, linear dose response and good stability when stored in environmental conditions. Therefore, deficiencies such as fading, low sensitivity and poor humidity are not seen in them. In addition, lithium borate crystals doped with different elements have the ability to detect neutrons due to the presence of Li and B, both of which have large neutron absorption capacities. During the past decades, a great deal of research has been focused on the synthesis and study the physical properties of borate compounds [10,11]. Generally, $\text{Li}_2\text{B}_4\text{O}_7$ (LTB) as a borate compound is used in the dosimetric application but it does not comply with all the requirements so studies are still being continued to improve its properties [12-18]. Over the past few years, attempts have been devoted to TL properties of nanomaterials because of their exceptional TL response compared with their bulk counterparts [19-36]. The basic problem with the conventional TL materials is saturation of trapping states at high absorbed doses, which causes the TL response to become sub-linear in high radiation exposure. A general feature of nanostructures is that they exhibit linear dose response at higher exposures, where the known micro-scaled TL materials saturate [37]. LTB is one of the important nano-crystals that is produced in different shapes and sizes to improve its optical properties in a variety of techniques [37-38]. However, the TL properties of LTB: Cu-Mg nanoparticles (NPs) have not been reported so far. This work deals with the synthesis procedure of LTB-NPs co-

doped with different percentage of Cu and Mg via the combustion method. X-Ray diffraction patterns (XRD) along with scattering electron microscopy (SEM) were used to determine the structure, shape and size of NPs. In the following, the optical properties of LTB-NPs including the UV-visible spectrum as a tool to estimate the optical band gap as well as TL dosimetry features under gamma irradiation for personal dosimetry application are presented.

2. MATERIALS AND METHODS

LTB-NPs co-doped by Cu and Mg impurities were synthesized by the solid-state reaction route. All materials used in this work were produced from Merck Corporation with high purity. Firstly, 0.696 g of Lithium nitrate, 0.925 g of Boric acid, 4.082 g of Ammonium nitrate and 3.062 g of Urea were mixed and pulverized for 5 min. Then, the mixture was placed in a furnace at different temperature (380° C, 480°C, 580° C) for one hour. Subsequently, productions were crushed for 10 min. Finally, the nano-powder was annealed at 450 °C for 10 minutes in a crucible and was quenched by taking the crucible out of the furnace and placing it on a metal block. Various amounts of Copper nitrate and Magnesium nitrate were added to the starting mixture for producing LTB-NPs doped with Cu and Mg.

The structure of the powders was specified via XRD at room temperature by using Cu-target (Cu-K α line, $\lambda=1.54056$ Å) with Rigaku D-maxc III diffractometer. The shape and morphology were specified via SEM (Model KYKY-EM3200). The UV–visible absorption spectrum was recorded by using Shimadzu UV1700 spectrometer. All the irradiations were carried out using ^{60}Co source at the secondary standard Dosimetry Laboratory (SSDL) at Karaj-Iran. TL glow curves were recorded at a TLD reader model Harshaw 4500 by the contact heating with the heating rate of 1K/sec.

3. RESULTS AND DISCUSSION:

The SEM images of the samples synthesized by combustion method at 380, 480 and 580° C are shown in figure 1a, b and c respectively.

Fig 1. SEM images of LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) synthesized by combustion method at different temperatures of a)380° C b)480°C c)580° C.

As is observed, at 380° C, the bulk material is clinging and with increasing the temperature to 480° C the particle size become smaller and the composition of spherical and rod nanoparticles are formed. At the temperature of 580° C nanoparticles are produced which refers to the process of burning and complete composition of raw materials in the reaction. The structure of the produced LTB was investigated by XRD patterns, which is shown in figure 2.

Fig. 2. XRD patterns of LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) synthesized via combustion method at
a) 380° C b) 480° C c) 580° C.

According to the figure 2 (a) and (b), the XRD pattern of the samples synthesized at temperatures below 580° C show several additional peaks due to existence of crystalline phases other than LTB which could be produced by incomplete reactions between the starting materials. Therefore, the synthesized sample at temperature 580° C (LTB-NPs) was selected for further analysis. XRD peaks are compared with the original LTB card using the International Center for Diffraction Data (ICDD) PDF card (ICDD 40-0505 (LTB)) for phase identification. XRD pattern of LTB-NPs confirmed tetragonal crystalline phase of the sample and the sharpens of the peaks in figure

2c confirms the good crystallinity. Williamson-Hall's formula determines the crystallite size of the NPs through the below relation [39]:

$$\beta \cos \theta = (0.89 \lambda) / d + 4 \epsilon \sin \theta \quad (1)$$

where λ , β , θ , ϵ and d respectively ascertain the wavelength of Cu K α radiation (1.5406 Å), full-width at half maximum of the diffraction peak (FWHM), Bragg angle, strain and the mean size of particles [39]. Figure 3 shows the variation of $\beta \cos \theta$ with $4 \sin \theta$. According to the linear fit of the data, the crystallite size is determined from the y-intercept using Eq. 1. The average size of LTB-NPs was obtained as ~ 47 nm.

Fig.3. Williamson-Hall's plot of LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) NPs synthesized via combustion method.

The absorbance spectrum of LTB-NPs is depicted in Figure 4 with the main peak around 290 nm. The inset graph shows the Tauc plot which can be used to estimate the band gap through the below equation [40]:

$$\alpha h\nu = A (h\nu - E_g)^n \quad (2)$$

Where A is a constant, $h\nu$ is photon energy, α is the absorption coefficient, E_g is the optical band gap, $n = 1/2$ for the direct transition and $n = 2$ for indirect transition. The linear fit of $(\alpha h\nu)^2$ vs. $h\nu$ allows calculating the band gap of the LTP-NPs. The indirect optical band gap of ~ 3.7 eV was measured for the synthesized NPs from the UV

visible absorption spectra by using the Tauc plot (inset of the figure) and identifying the intercept at abscissa, which is in accordance with the previous work [41].

Fig. 4. Absorption spectra LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) NPs. The inset shows the Tauc plot. The main purpose of this study is to investigate the TL properties of LTB crystals doped with Cu and Mg which have the highest sensitivity to gamma radiation. The sample synthesized at temperature of 380° C did not show any sensitivity to gamma rays and the sample synthesized at 580° C was 2 times more sensitive to gamma rays than that produced at 480° C. Therefore, after examining the sensitivity of the 3 samples mentioned above, nanopowder samples (synthesized at 580° C) with the highest sensitivity to gamma rays and high crystalline grade compared to the other two samples were selected for TL dosimetry of gamma rays. However, the increased sensitivity to gamma rays in nanopowders synthesized at higher temperatures can be attributed to good replacement of impurities in the crystal structure.

The effect of Cu and Mg impurities on the TL sensitivity of LTB-NPs is also studied. At first, the amount of copper was kept constant (0.02 wt%) then Mg was used with amounts of 0.01, 0.03, 0.05, 0.1, 0.5 and 1 wt%. Subsequently, Mg was taken constant (0.5 wt%) and various concentration of Cu (0.01, 0.03, 0.05, 0.1, 0.5 and 1 wt%) was added to the mixture. TL features of the produced LTB:Cu,Mg NPs were investigated following irradiation with the ^{60}Co gamma ray at room temperature. Figure 5 illustrates the effect of Cu and Mg impurities on the TL response of LTB nanopowders.

As is showed in figure 5, 0.02 wt % of Cu and 0.5 wt % of Mg are the optimal impurity concentrations in LTB-NPs to achieve the highest TL sensitivity.

In this study, to investigate the TL glow curve, the general order kinetics model, which is widely used to describe the TL peaks, was used. A computerized glow curve deconvolution (CGCD) technique was applied to estimate the trapping parameters of the overlapping peaks based on this model. In this method, a glow curve deconvolution function is exploited in terms of the intensity and the temperature of the peak maximum (I_m , T_m), kinetic order (b) and activation energy (E) where the equation of TL intensity is as follows [42]

$$I(T) = I_m b^{\frac{b}{b-1}} \exp\left(\frac{E}{kT} \cdot \frac{T - T_m}{T_m}\right) \times \left[(b-1)(1-\Delta) \frac{T^2}{T_m^2} \cdot \exp\left(\frac{E}{kT} \cdot \frac{T - T_m}{T_m}\right) + Z_m\right]^{\frac{-b}{b-1}} \quad (3)$$

Where k and T are the Boltzmann's constant and temperature respectively and $\Delta = 2kT / E$ (4)

$$\Delta_m = 2kT_m / E \quad (5)$$

$$Z_m = 1 + (b-1)\Delta_m \quad (6)$$

Figure 6 and Table 1 show the component glow peaks and the TL kinetic parameters of the LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) NPs irradiated with ^{60}Co gamma rays.

A relatively low figure of merit (FOM) of 1% indicates good agreement of experimental and theoretical peaks based on the general order of the kinetic model [43].

Fig.6. Experimental and deconvoluted glow curve of LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) NPs for 30000 Gy gamma irradiation with ^{60}Co source.

Table 1: TL trapping parameters of component glow peaks of LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) NPs.

peaks	LTB: Cu, Mg (0.02 % wt Cu and 0.5 % wt Mg) NPs			
	$I_m(\text{a.u})$	$T_m (\text{K})$	$E (\text{eV})$	b
a	17000	445	0.97	1.2
b	1000	473	0.69	1
c	502	493	1.67	1.2
d	11722	541	0.91	1.3
e	2690	618	1.68	2

As is observed in figure 6, the glow curve contains five overlapping glow peaks between 445 and 618 K. The glow curve of the sample has two major peaks around 445 and 541 K and 3 small satellite peaks around 473, 493 and 618 K. The TL properties of materials are highly dependent on the natural defects and the impurities inserted

in the host lattice [44]. Based on the previous reports, Cu^+ ions not only substitute for Li^+ ions but also situate at the interstitial sites and act as charge compensator in the LTB structure [45]. After irradiation, Cu^+ ions act as recombination or trapping centers and become Cu^{2+} active or Cu_A^0 and Cu_B^0 form respectively [45,46]. The glow peak observed near 445 K might be attributed to the decay process of Cu during heating. It is worth noting that the relative intensity of TL is changed with increasing Mg concentration. Some of Li atoms are in the tetrahedral lacunas which have little aberrance, and the others are in the oxygenic octahedral lacunas which have much aberrance [47]. When the dopant atoms with a large radius come into the LTB structure, the lattice will be distorted. The length of the B-O bond in some complexes (BO_3) possibly decreases. The lacunas which contain the dopant atoms are not tetrahedrons or octahedrons any longer, the number of oxygenic atoms that bond with the dopant atom can reach 7 or 8, and the bond distances have a large range. When the Mg dopant atoms come into the LTB structure, the complexes MgO and B_2O_3 are generated; especially the latter. This has some relations with the traps corresponding to the TL glow peak in this phosphor [48].

In the following, the TL dose response of NPs under gamma irradiation is investigated. LTB-NPs samples were irradiated in the absorbed dose range of 10 Gy to 30 KGy. Figure 7 shows the dose response of LTB-NPs doped with Cu- Mg on a logarithmic basis. The dose response is defined as the functional dependence of the intensity of the measured TL signal upon the adsorbed dose.

Fig.7.Dose response of LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) NPs irradiated with ^{60}Co source (30000 Gy).

LTB nanoparticles doped with Cu-Mg have a linear behaviour in the range of 500 Gy to 30000 Gy. To determine the fading of LTB-NPs, the nanopowders were firstly irradiated to gamma radiation (30000 Gy) at room temperature and then were kept at dark at room temperature. Afterwards, the TL response was measured at different storage times up to 30 days. Figure 8 shows the fading curve for nanocrystalline doped with Cu-Mg impurities. As is evident, the stored TL signal experiences about 10% fading after one month of storage at room temperature and then remains unchanged.

Fig 8. TL response of LTB: Cu, Mg (0.02 wt % Cu and 0.5 wt % Mg) NPs irradiated with ^{60}Co source (30000 Gy) after 30 days.

4. CONCLUSION:

In this research LTB-NPs co-doped with Cu and Mg were synthesized via combustion method. The experimental results show that the nanopowders could be produced at 580 °C for 1 hour at ambient pressure. Under these conditions, the LTB-NPs with an average size of ~47 nm and tetragonal phase as well as the optical band gap of ~ 3.7 eV are produced. The annealed LTB-NPs co-doped by 0.02 wt % Cu and 0.5 wt % Mg and irradiated with γ -rays exhibits a TL glow curve including five overlapping peaks. In the present study, the fading characteristic was also examined which is one of the most important TL characteristics. It was found that almost 10% of the stored TL fades after one month which may be due to the low temperature peak (first peak) of Cu-Mg doped LTB. The TL response of the sample increases linearly with increasing gamma-ray dose between 500 Gy to 30000 Gy.

Due to the effective atomic number of LTB, which fairly matches the body tissue as well as high sensitivity, the linearity of dose response in a wide range of absorbed dose and low fading, synthesized nanoparticles co-doped with Cu and Mg is recommended as a suitable dosimeter for high dose dosimetry.

5. Acknowledgements:

The authors acknowledge the financial support of the Research Council of Persian Gulf University and Research Council of the University of Kashan.

6. Conflict of interest

The authors declare no competing financial interest.

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