

Research Article

Color Centers Formation in Lithium Fluoride Nanocubes Doped with Different Elements

Numan Salah,¹ Saeed S. Babkair,² and Ameer Azam¹

¹ Center of Nanotechnology, King Abdulaziz University, Jeddah 21589, Saudi Arabia

² Center of Nanotechnology, Department of Physics, Faculty of Science, King Abdulaziz University, Jeddah 21589, Saudi Arabia

Correspondence should be addressed to Numan Salah; nsalah@kau.edu.sa

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Nanocrystalline cubes of pure and doped LiF material were fabricated. They were doped with Cu, Ag, Dy, Tb, and Eu and studied for their PL properties. Shape of the obtained nanocubes was found to be modified by introducing dopants into the host of LiF. The crystallinity was also decreased by increasing the concentration of these dopants (i.e., Eu and Tb). These impurities could induce exothermic peaks at around 250°C in the measured DSC curves. Moreover, incorporating such impurities into the host of LiF was found to enhance intensity of the broad band at 370–550 nm that was observed in the pure one. Extra sharp emissions were also observed in Eu and Tb doped samples. These results showed that the active color centers created in pure LiF nanocubes can be enriched/enhanced by these impurities, mainly Eu and Tb. This implies that these nanocubes might be useful in the development of optical devices and advanced color center laser.

1. Introduction

Lithium fluoride (LiF) single crystal as well as doped with proper activators is a highly sensitive phosphor used in several applications such as integrated optics, color center laser, and radiation dosimetry. It has the highest UV transmission of all materials and is used for X-ray monochromator plates where its lattice spacing makes it the most suitable analysis crystal. It has low effective atomic number (8.14), which is sufficiently close to that of the biological tissue (7.4). This material is sensitive to ionization radiation, especially when doped with proper activators. LiF doped with Mg, Cu, and P is found to be the most sensitive material for ionizing radiations using thermoluminescence (TL) technique. This material was first synthesized and reported in 1978 by Nakajima et al. [1]. Thereafter, many research groups produced the phosphor using different procedures and studied its characteristics [2–7]. Other workers have investigated the role of impurities in the TL of LiF:Mg, Cu, and P phosphor [6–11]. The nanostructure form of LiF:Mg, Cu, and P was prepared for the first time by Salah et al. [12] and studied for its TL response. They found that this material in nanoform is

sensitive to gamma rays in a wide range of exposures (0.1 Gy–10 kGy).

Color center is a type of crystallographic defect in which an anionic vacancy in a crystal is filled by one or more electrons. In fact, this depends on the charge of the missing ion in the crystal. When a compound is heated to high temperature, the electrons get excited and get displaced from their respective positions in the crystal structure leaving behind some electrons in the vacated spaces. This leads to the phenomenon of coloring of compounds. Formation of color centers in LiF by irradiation was reported and studied in detail by several authors [13–17]. However, reports on formation and modification of color centers in LiF without irradiation rarely appeared. Such color centers were also not studied in the nanostructure form of LiF. It has been reported that the large specific surface area of nanostructured materials can increase the absorption and then the quantum yields [18]. Recent study by Alharbi et al. [19] showed an excellent property in LiF nanostructure, which is the formation of a broad band at around 370–550 nm without irradiation. This band was attributed to the color centers induced in the host of LiF due to oxygen defects, which might be created during

the synthesis process. They have produced size-controlled nanocubes of pure and silver doped LiF by the chemical coprecipitation method. Doping this material with Ag ion was found to enhance the PL property of LiF nanocubes. Therefore, it is very interesting to further study the effect of other impurities such as rare earths and other elements on the PL property of this important material.

In this work, we report on producing nanocrystalline cubes of pure and copper (Cu), silver (Ag), dysprosium (Dy), terbium (Tb), and europium (Eu) doped LiF by the chemical coprecipitation method. The as-synthesized samples were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), and differential scanning calorimetry (DSC) and studied for their photoluminescence (PL) properties. The results show that the shape and crystallinity of these nanocubes were modified by introducing dopants into the host of LiF. These impurities were found to enhance intensity of the broad band of the pure sample. Extra sharp emissions were also observed in Eu and Tb doped samples. These results showed that the active color centers created in pure LiF nanocubes can be enriched/enhanced by these impurities, mainly Eu and Tb. This suggests that these nanocubes might be useful in several applications such as optical devices and color center laser.

2. Experimental Details

Nanocrystalline cubes of pure and copper (Cu), silver (Ag), dysprosium (Dy), terbium (Tb), and europium (Eu) doped LiF were produced by the chemical co-precipitation method as adopted earlier by Alharbi et al. [19]. They were synthesized using AR grade chemicals at room temperature. Water and ethanol were used as solvents at a ratio of 0.1:1. Initially, the desired concentration of lithium chloride (LiCl) was dissolved in triply distilled deionized water. The normality of the solution was 0.2 N. Then, this solution was mixed with ammonium fluoride (NH_4F) solution (0.2 N) stoichiometrically. The solution of NH_4F was added to the solution of LiCl drop wise with continuous stirring. The precipitate was filtered out and washed several times with distilled water. The powder samples, thus obtained, were dried at 343 K in an oven for 2 h. The used impurities in this study were added in their chloride forms. A typical concentration of these impurities, that is, 0.5 mole% was used in LiF samples except those doped with Eu and Tb, where samples with different concentrations of dopants were obtained. These concentrations are 0.1, 0.2, 0.5, and 1 mole%. In a typical case, the desired concentration of the impurity, that is, $\text{DyCl}_3 \cdot 6\text{H}_2\text{O}$ was added to the solution of LiCl and stirred for 1 h before adding the solution of NH_4F as mentioned above.

The as-synthesized LiF powder samples were characterized by XRD, using an Ultima-IV (Rigaku, Japan) diffractometer with Cu $K\alpha$ radiation, while the morphology of these samples was studied by SEM using a field emission scanning electron microscopy (FESEM), JSM-7500 F (JEOL-Japan) operated at 15 kV. Photoluminescence (PL) emission spectra were recorded at an excitation wavelength of 325 nm using a fluorescence spectrofluorophotometer, model RF-5301 PC, Shimadzu, Japan. The measurement was performed

at room temperature. The thermal behavior in pure LiF and Eu, Tb doped samples was studied under nonisothermal measurements by using a Shimadzu DSC-60 instrument. Typically, 5 mg of sample in powder form was sealed in standard aluminum sample pans and heated at a heating rate of $10^\circ\text{C}/\text{min}$. The temperature precision of this equipment is $\pm 0.1\text{ K}$.

3. Results and Discussion

SEM images of the as-synthesized pure and Eu doped LiF powder samples are presented in Figures 1(a)–1(e). The pure sample has nanocubes with sizes in the range of 40–90 nm (Figure 1(a)). This figure shows that the shapes of these nanocubes were slightly modified by introducing Eu as impurity into the LiF host (Figures 1(b)–1(e)). This modification is clearly observed in shape of Eu doped samples by increasing the concentration of this impurity from 0.1 to 1 mole%. The modification includes a loss in uniformity of the nanocubes' structure and showing irregular shapes. Moreover, with increasing the concentration of Eu in LiF, host small shiny nanorods are formed. These nanorods are clearly observed at higher concentrations of Eu as shown in Figure 1(e). Similar trend is also observed in the Tb doped samples (Figures 2(a)–2(d)), mainly the formation of irregular shapes. These modifications in shapes of these nanocubes by adding impurities, that is, Eu and Tb might be due to the formation of new phases or other compounds. It is possible that an interruption in the crystal structure of LiF is occurred by inducing the dopant ions into the host and causing irregularity in these crystals. Moreover, small portion of these doped crystals might be able to form rod structures instead of cubes as shown in Eu doped samples (Figure 1(e)). The effect of an impurity in the nanoscale crystals is expected to be different than that in a single big crystal (in the bulk or microsize). In the nanoscale crystals or nanocrystalline, the effect will be more visible.

XRD patterns of the as-synthesized pure and Eu doped LiF samples are shown in Figure 3 (curves (a), (b), (c), (d), and (e)). The XRD result of pure sample (curve (a)) shows several diffracted peaks with hkl values indicating a complete crystalline structures in a griceite phase. Moreover, there is significant broadening in the XRD peaks, which implies that the samples are nanosized (in cubic forms). This result is similar to that reported earlier by Alharbi et al. [19]. However, the crystallinity of these nanocubes decreases by inducing Eu as a dopant in the host of LiF (Figure 3, curves (b), (c), (d), and (e)). This crystallinity systematically decreases by increasing the concentration of Eu. Furthermore, by inducing Eu into the host of LiF, small diffracted peak is observed at around $2\theta = 33^\circ$. This peak might be due to the formation of small amount of europium oxide (DB card number 00-046-0427) (Figure 1(e)). Similarly, Tb doped samples show almost the same trend, where oxide compound (or other compound) of terbium might get formed (Figure 4 curves (a), (b), (c), (d), and (e)). The result shown in XRD study is in agreement with that shown in SEM images. The formation of oxide compounds of these impurities might be the reason for forming small shiny nanorods.

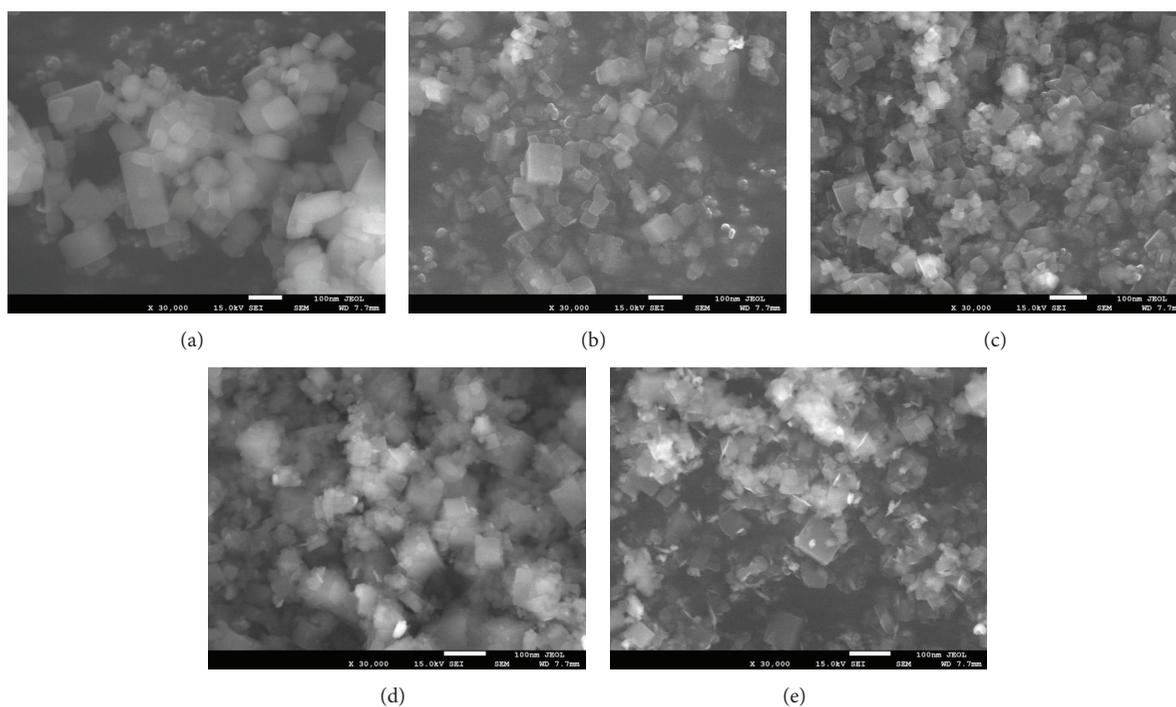


FIGURE 1: SEM images of the as-synthesized nanocubes of (a) pure LiF and Eu doped samples at different concentrations: (b) 0.1 mole%, (c) 0.2 mole%, (d) 0.5 mole%, and (e) 1 mole%.

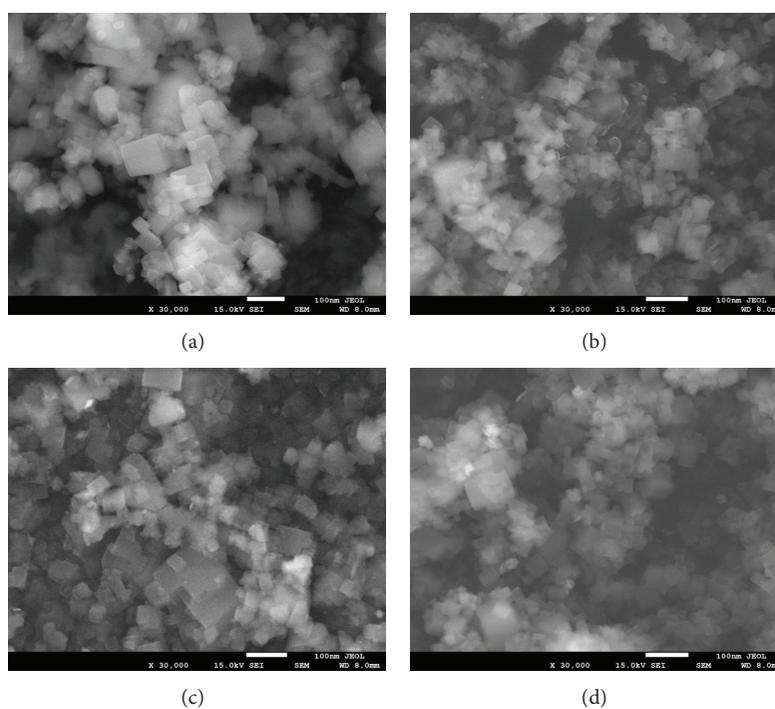


FIGURE 2: SEM images of the as-synthesized nanocubes of LiF doped with Tb at different concentrations: (a) 0.1 mole% (b) 0.2 mole%, (c) 0.5 mole%, and (d) 1 mole%.

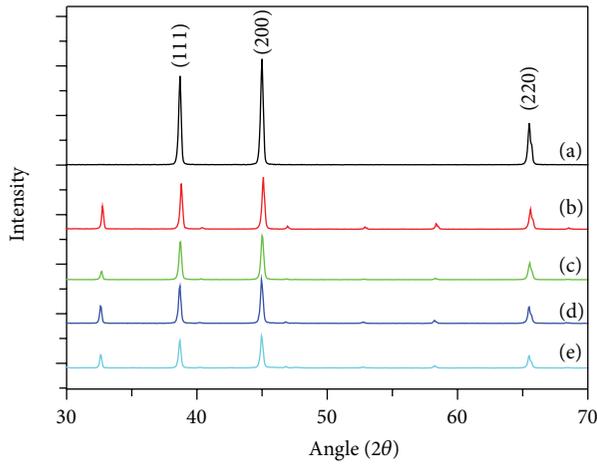


FIGURE 3: XRD pattern of the as-synthesized nanocubes of (a) pure LiF and Eu doped samples at different concentrations: (b) 0.1 mole%, (c) 0.2 mole%, (d) 0.5 mole%, and (e) 1 mole%.

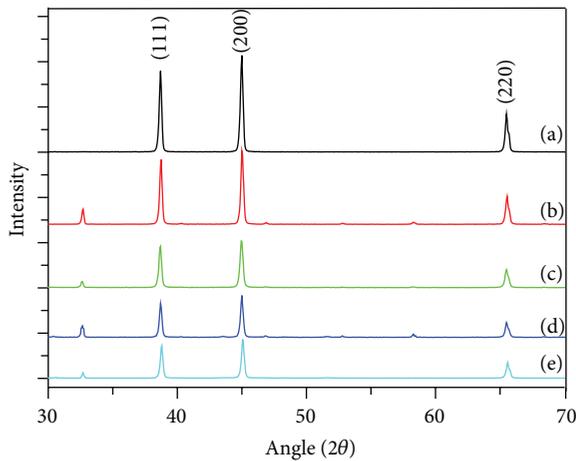


FIGURE 4: XRD pattern of the as-synthesized nanocubes of (a) pure LiF and Tb doped samples at different concentrations: (b) 0.1 mole%, (c) 0.2 mole%, (d) 0.5 mole%, and (e) 1 mole%.

Differential scanning calorimetry (DSC) measurements for pure and Eu and Tb doped samples at typical concentrations are shown in Figure 5 (curves (a), (b), and (c), resp.). The DSC curve of pure LiF nanocubes (curve (a)) has no endo- or exothermic peaks. This is a clear indication that pure LiF has only a single phase. The samples doped with Eu and Tb show one prominent exothermic peak at around 250°C. The existence of this peak in the doped samples might be of the oxide structure of the used impurity as mentioned above. This oxide structure has different crystal structure rather than cubes. This result corroborates the result obtained by SEM and XRD.

Figure 6 shows the PL emission spectra of pure and Cu, Ag, Dy, Tb, and Eu doped LiF nanocubes, curves (a), (b), (c), (d), (e), and (f), respectively. The emission spectrum of pure sample (curve (a)) shows a broad band in the range of 370–550 nm. This band might be assigned to the F_2^- and F_3^{2-} color centers (two electrons captured by two and three anion vacancies, resp.) [19]. These centers could be induced in the

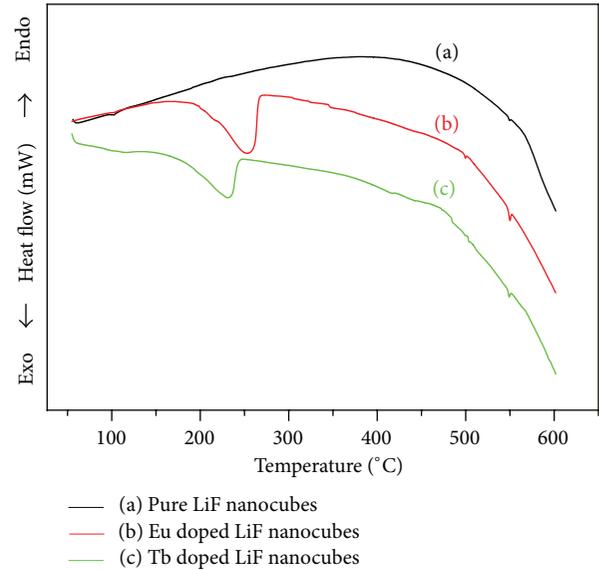


FIGURE 5: DSC plot for the as-synthesized nanocubes of LiF: (a) pure, (b) Eu doped, and (c) Tb doped samples at concentrations of 0.5 mole%.

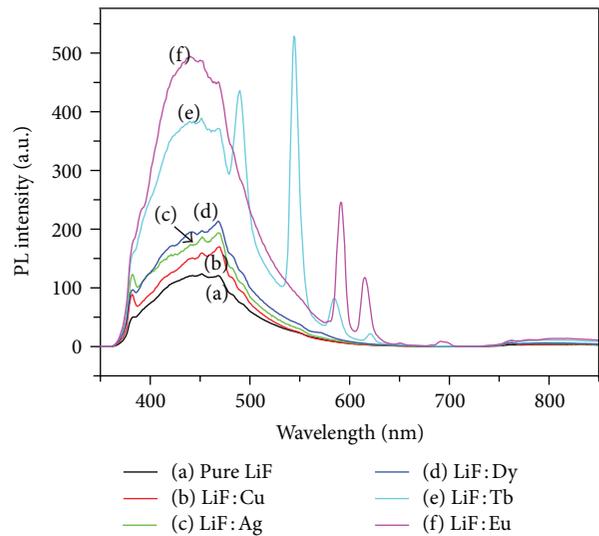


FIGURE 6: PL emission spectra of the as-synthesized nanocubes of pure and doped LiF samples with different impurities at a concentration of 0.5 mole%.

host of LiF due to oxygen defects, which might be created during the synthesis process. It has been reported [20] that oxygen defects/contaminations can lead to such emissions, but at the higher wavelength side of the visible region. In the present LiF nanocubes, the shift in the emission band to the lower wavelength might be due to widening in the band gap of the material as a result of reducing the particle size to the nanoscale.

The emission spectra of Cu, Ag, and Dy doped samples (Figure 6, curves (b), (c), and (d), resp.) are almost similar to that of the pure one (Figure 6, curve (a)), but with further

PL enhancement. The PL emission spectra of Tb and Eu doped samples (Figure 6, curves (e) and (f)) show strong enhancement in intensity of the broad band at 370–550 nm, with the emergence of extra sharp emissions. The emission spectrum of Tb doped sample shows two strong sharp emissions peaking at 488 and 544 nm along with two smaller ones at around 585 and 620 nm. These bands are the well-known emissions of Tb^{3+} ions. They can be assigned to the $^5D_4 \rightarrow ^7F_6$, $^5D_4 \rightarrow ^7F_5$, $^5D_4 \rightarrow ^7F_4$, and $^5D_4 \rightarrow ^7F_3$ transitions of Tb^{3+} ion, respectively [21–23]. In the Eu doped sample, the broad band at 370–550 nm has the highest PL intensity in association with two bands located at 590 and 615 nm, which are the well-known emissions of Eu^{3+} ion [23]. The bands at 590 and 615 nm are due to $^5D_0 \rightarrow ^7F_1$ and $^5D_0 \rightarrow ^7F_2$ transitions of Eu^{3+} ion, respectively. Small hump at around 700 nm can also be seen, which can be attributed to $^5D_0 \rightarrow ^7F_3$ transition of Eu^{3+} ion. Ag doped LiF sample was studied earlier [19], but at different concentrations of Ag. The PL of this sample showed prominent emission at 420 nm, which has been enhanced by increasing the concentration of Ag ions. The optimum value for the concentration of Ag to show maximum PL emission was found to be 0.08 mole% [19]. This band could be assigned to the contribution from the surface resonance band of Ag [24, 25]. In the present work, a typical concentration of 0.5 mole% for Ag ions was selected and included in this study for comparison propose, since the optimum values for other impurities were unknown.

The observed enhancement in intensity of the broad band at 370–550 nm by introducing impurities into the host of LiF (Figure 6) might be due to overlapping of the energy levels (color centers) of oxygen defects with those created by these impurities inside the band gap of LiF. Normally, Ag and Cu ions as impurities show their emissions at the blue region of the visible region [24–26]. In the present case, these emissions will overlap with that of oxygen defects leading to further enhancement. Dysprosium ion might get introduced into the host of LiF matrix in its 3+ form (Dy^{3+}) and this ion is a well-known activator mostly showing its emission in the visible region. This ion was reported by several authors to have two emissions at around 485 and 572 nm [27]. In the present Dy doped LiF sample, the first band might be included within the broad band at 370–550 nm, while the second one is shown as a small hump at around 570 nm (Figure 6, curve (d)). Copper ions might have been incorporated in their 1+ form (Cu^+) as reported earlier by Patil and Moharil [6], but as mentioned above the emission will be in the blue region [26]. Therefore, this emission might get included within the broad band at 370–550 nm and thus could show further PL enhancement.

PL emission spectra of pure LiF nanocubes and Eu doped samples (at different concentrations) are presented in Figure 7, curves (a), (b), (c), (d), and (e), respectively. This figure shows that the optimum value for the concentration of Eu in LiF host is 0.5 mole%. At this concentration, the intensity of the broad band at 370–550 nm is increased by a factor of approximately 4 compared with that of the pure sample. Beyond this concentration, the intensity decreases, which might be due to the PL quenching. This quenching normally occurs at the higher concentrations of dopants [28].

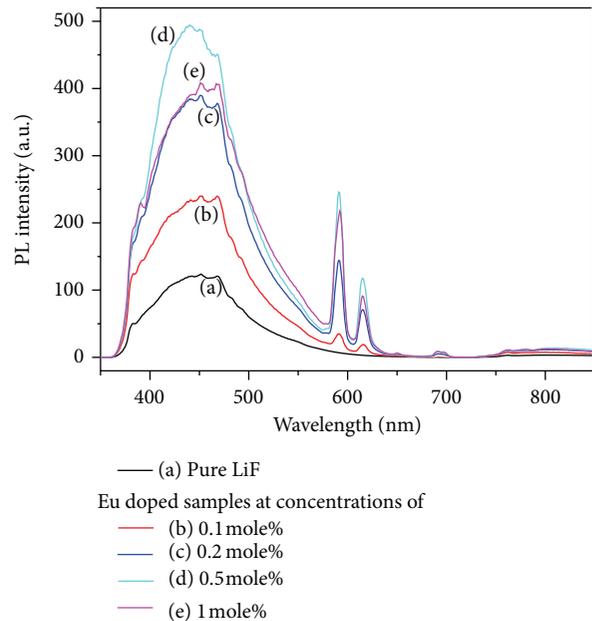


FIGURE 7: PL emission spectra of the as-synthesized nanocubes of pure and Eu doped LiF samples at different concentrations.

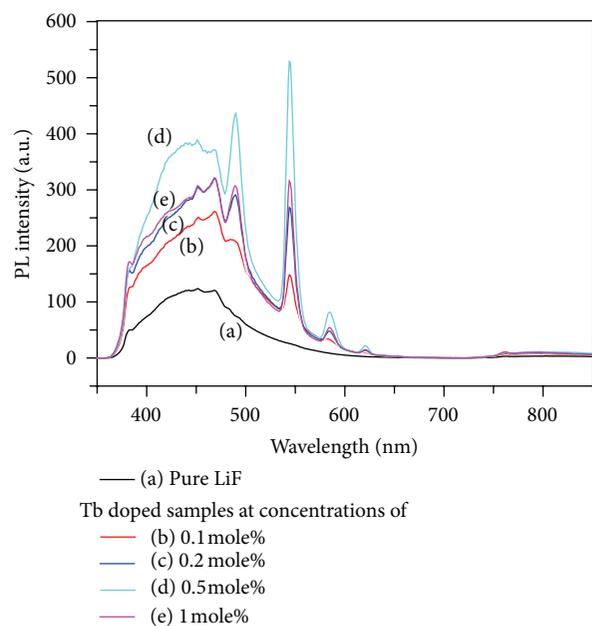


FIGURE 8: PL emission spectra of the as-synthesized nanocubes of pure and Tb doped LiF samples at different concentrations.

Similarly, Tb ions show maximum emissions at a concentration of 0.5 mole% (Figure 8). The sharp band at 540 nm in Tb doped sample (at a concentration of 0.5 mole%) is strongly enhanced suggesting that this nanomaterial might have potential application as a monochromatic laser material.

From the application point of view, the results presented in this study show that pure nanocubes of LiF have active color centers without irradiation, which could be

enriched/enhanced by Eu and Tb dopants. This implies that these nanocubes might be useful in the development of optical devices and advanced color center laser. Moreover, the method of producing this material in a nanoscale size is very simple and nonexpensive. These nanostructures are of great advantage due to the increase in the specific surface area, which is responsible for increasing the absorption and then the quantum yields.

4. Conclusions

In this work, we reported on fabricating nanocrystalline cubes of pure and doped LiF material. They were doped with Cu, Ag, Dy, Tb, and Eu and studied for their PL properties. Shape of the obtained nanocubes was found to be modified by introducing dopants into the host of LiF. The crystallinity was also decreased by increasing the concentration of these dopants, that is, Eu and Tb. These impurities could induce exothermic peaks at around 250°C in the measured DSC curves. Moreover, incorporating such impurities into the host of LiF was found to enhance intensity of the broad band at 370–550 nm that was observed in the pure one. Extra sharp emissions were also observed in Eu and Tb doped samples. The optimal concentrations of Eu and Tb in LiF host for maximum emissions were found to be 0.5 mole%. These results showed that the active color centers created in pure LiF nanocubes can be enriched/enhanced by these impurities mainly Eu and Tb. This implies that these nanocubes might be useful in the development of optical devices and advanced color center laser.

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