

Research Article

Luminescence Properties of CaF_2 Nanostructure Activated by Different Elements

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Nanostructures of calcium fluoride (CaF_2) doped with Eu, Tb, Dy, Cu, and Ag were synthesized by the coprecipitation method and studied for their thermoluminescence (TL) and photoluminescence (PL) properties. The PL emission spectrum of pure CaF_2 nanostructure has a broad band in the 370–550 nm range. Similar spectra were observed in case of doped samples, beside extra bands related to these impurities. The maximum PL intensity was observed in Eu doped sample. The TL results of Eu, Cu, Ag, and Tb doped samples show weak glow peaks below 125°C, whereas Dy doped one is found to be highly sensitive with a prominent peak at 165°C. This sample was further exposed to a wide range of gamma rays exposures from ^{137}Cs source. The response curve is linear in the 100 Gy–10 kGy range. It is also observed that the particle size of CaF_2 nanostructure was significantly reduced by increasing Dy concentration. These results showed that Dy is a proper activator in the host of CaF_2 nanostructure, providing a highly sensitive dosimeter in a wide range of exposures and also plays a role as a controlling agent for particle size growth.

1. Introduction

Nanoscale materials or nanostructures have attracted huge attention in the last two decades due to their unique properties. They have a potential to be used in a variety of applications. A large number of individuals and research groups from different fields have produced different nanomaterials and studied their properties. These include structural, optical, electrical, magnetic, mechanical, and dosimetric properties [1–5]. Many methods of preparations have also been developed in the last two decades, where different nanostructures like nanoparticles, nanocubes, nanowires, nanorods, and so forth, of several materials have been produced [6, 7]. Recent investigations have showed that the optical, luminescent, and other properties can be modified by the shape and size of the nanostructures. The role of impurity(ies) in the host of these nanostructure is another parameter that can be used to modify their properties [8–10].

Thermoluminescence (TL) is a well-known technique widely used for detection and measurement of absorbed radiation and dating of archaeological samples. It is a powerful technique for detecting defects in solid materials and dosimetry of different ionizing radiations. The presently used TL dosimeter (TLD) materials are inorganic crystalline materials mainly low Z effective compounds. However, the selected TLD materials have advantage and drawbacks; therefore, efforts are still going on to improve their dosimetric properties. These improvements were tried to be achieved by either improving the TL characteristics of these TLD materials by preparing them using different methods or by doping with different impurities [11, 12]. Almost all of these phosphors have “dose ranges” depending on their “TL sensitivity” and “response characteristics” (linearity and saturation) to high energy radiations.

In the last few years, Salah and his group have produced nanostructures of some highly sensitive phosphors [13–21]

and studied their TL response to different ionizing radiations. They observed that these nanostructures have unique dosimetric properties, mainly their linear response over a wide range of exposures along with a negligible fading. These nanomaterials are insensitive to heat treatments, making them quite useful to estimate the heavy doses of ionizing radiations. These results are observed in tissue and nontissue equivalent nanomaterials [13–21]. Other workers have also participated in testing TL response of some nanostructure materials to different ionizing radiations [22–28]. These studies showed that the TL response of these nanomaterials to ionizing radiations is mostly linear in a wide range of exposures; however, few studies were focused on the TL response of low Z effective nanomaterials.

Calcium fluoride (CaF_2) is a wide band gap material with a large-scale transparency. Therefore, color center formation is possible just by irradiating CaF_2 by ionizing radiation [29]. The material has a relatively low Z effective, making it suitable for ionizing radiations used in radiotherapy. It was reported that this material is suitable as a laser material particularly, when doped with rare earth elements [30]. The nanostructure form of CaF_2 was synthesized by different methods [29, 31–33]. However, the TL and PL properties of this nanomaterial have not been well studied. Few studies were focused on the effect of different dopants on its optical properties [34–36]. Other investigations were focused on different routes for the syntheses of CaF_2 nanocrystals and study of the upconversion luminescence by doping with some rare earths [37, 38]. In this work CaF_2 nanostructure particles were doped with different impurities and studied for their TL response to gamma rays in a wide range of exposures. The effect of dopant concentration on the particle shape and size of CaF_2 was also investigated (i.e., Dy in CaF_2 nanocubes). Pure and doped samples were synthesized by the coprecipitation method. They were doped with Eu, Tb, Dy, Cu, and Ag. The as synthesized nanomaterials were characterized by XRD, SEM, DSC, and PL. Then they were exposed to a wide range of gamma rays exposures.

2. Experimental

Pure, europium (Eu), dysprosium (Dy), terbium (Tb), silver (Ag) and copper (Cu) doped CaF_2 nanocrystalline samples were synthesized by the chemical coprecipitation method. The samples of CaF_2 were synthesized by using water and ethanol as solvents at a ratio of 1:1. The desired concentration of calcium chloride (CaCl_2) was dissolved in triply distilled DI water. The normality of the solution was kept at 0.2 N. This solution was mixed with ammonium fluoride (NH_4F) solution (has a normality of 0.2 N). The solution of ammonium fluoride was added to that of calcium chloride dropwise with continuous stirring. The formed precipitate was filtered out and washed with distilled water several times. The resulting powder samples, thus obtained, were dried at 70°C in an oven for 3 hours. The used chemicals in this experiment are highly pure and were of AR grade. The dopants used in this study were incorporated in their chloride forms except that of Ag dopant, where nitrate compound is used. A typical

concentration of these impurities, that is, 0.2 mole%, is used in CaF_2 samples except those doped with Dy, where different concentrations in the range 0.05–2 mole% are studied. 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 CaCl_2 and stirred for one hour before adding the solution of NH_4F as mentioned above. For TL study the produced samples were annealed at 350°C for 1 h.

The samples of CaF_2 were characterized by X-ray diffraction (XRD), using an Ultima-IV (Rigaku, Japan) diffractometer with $\text{Cu K}\alpha$ radiation, while the morphology of these samples was studied by a field emission scanning electron microscopy (FESEM), JSM-7500 F (JEOL, Japan). Photoluminescence (PL) emission spectra were recorded at an excitation wavelength of 325 nm using a fluorescence spectrofluorophotometer, model RF-5301 PC, Shimadzu, Japan. A cutoff filter (UV-39) is used to block the emissions from the excitation source or scattered light. The PL measurement was conducted at room temperature. The study of temperature behavior 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 ± 0.1 K. Thermoluminescence (TL) glow curves were recorded on a Harshaw TLD reader (model 3500) under nitrogen atmosphere at a heating rate of $5^\circ\text{C}/\text{s}$. Neutral density filters of optimized density were used to avoid saturation of the photomultiplier tube (PMT) detector. For TL measurement 5 mg of sample was taken each time. The background reading is initially recorded and subtracted from the samples reading.

3. Results and Discussion

Figure 1 shows SEM images at different magnifications ((a) and (b)) of pure CaF_2 sample. These images show a mixture of spherical and cubic shape structures. These structures have sizes in the range of 20–80 nm. The produced nanostructures have a good particle size distribution. As mentioned in Section 2 that the used compounds of CaCl_2 and NH_4F were dissolved in water: ethanol mixture at a ratio of 1:1. This ratio was found to provide small nanostructures, while other ratios showed bigger particles.

Figure 2 shows XRD pattern of the as-synthesized pure CaF_2 sample. Several diffracted peaks can be seen with hkl values indicating a complete crystalline structure in a cubic phase (JCPDS Card number 87-0971). The displayed peaks correspond to values (1 1 1), (2 2 0), (3 1 1), and (4 0 0). The XRD pattern presents broad peaks revealing the small crystallite size of the synthesized samples. This result is similar to that reported in the literature [29]. The nanocrystalline size was calculated using Scherer's formula and found to be around 35 nm. This value is close to that observed by SEM (Figure 2). XRD of the doped samples was also studied, but the result is similar to that of pure CaF_2 nanostructure. The concentration of these dopants used in this study was low, which is 0.2 mole%. At this concentration

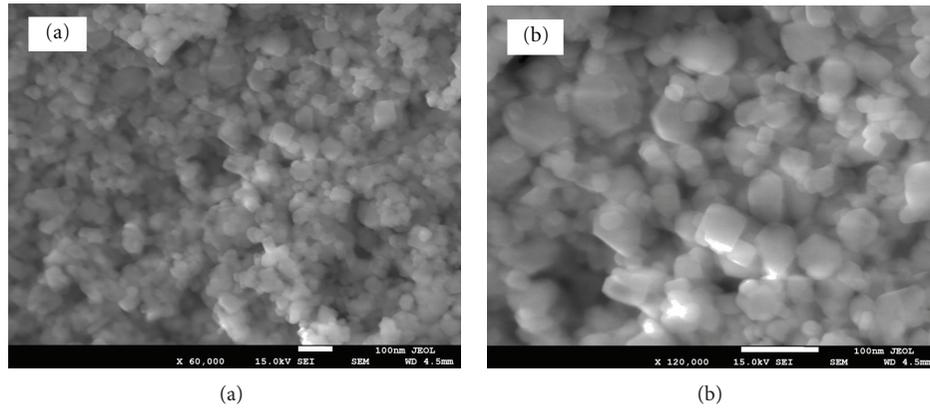


FIGURE 1: SEM images of the as-synthesized CaF_2 nanostructures taken at different magnifications.

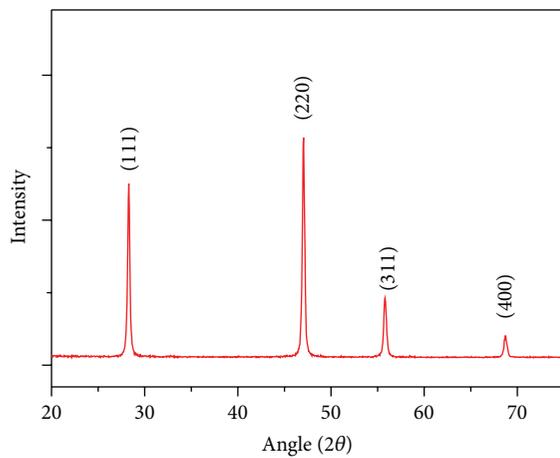


FIGURE 2: XRD pattern of the as-synthesized CaF_2 nanostructures.

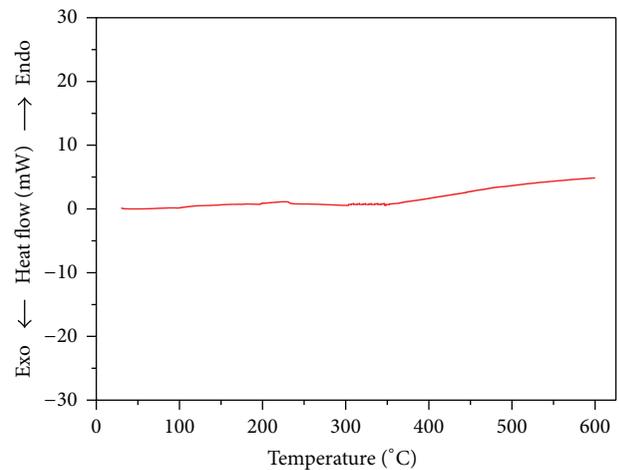


FIGURE 3: DSC plot for the as-synthesized CaF_2 nanostructures.

no significant changes are observed in the XRD peak of pure sample.

DSC measurement for pure CaF_2 sample was conducted in the range of 30–600°C. The DSC curve is shown in Figure 3. There is no endo- or exothermic peaks in this range, which means that pure CaF_2 has only a single phase. For the dosimetry using thermoluminescence technique, the material of the dosimeter should be thermally stable without any phase transitions in the range 40–400°C.

Figure 4 shows the PL emission spectra of the as-produced nanostructures of pure (curve (a)) and doped CaF_2 samples (curves (b)–(f)). As mentioned above, the samples of CaF_2 nanostructures were doped with Ag, Eu, Tb, Cu, and Dy at a concentration of 0.2 mole% and their PL result is shown in curves (b), (c), (d), (e), and (f), respectively. Pure sample (curve (a)) shows a broad band in the 370–550 nm range. This band might be induced due to the formation of color centers. These centers perhaps could be created by oxygen defects within the host of CaF_2 . It has been reported [39] that oxygen defects (contaminations) can induce such emission bands, but at the higher wavelength side of the visible region. In the present CaF_2 nanostructures, reducing the particle size

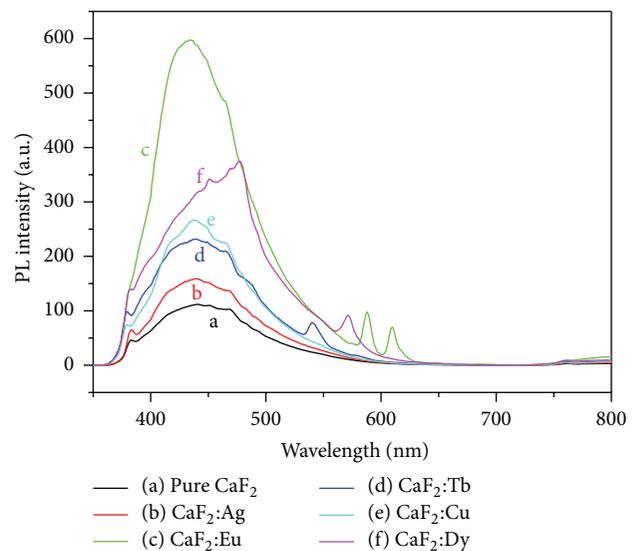


FIGURE 4: PL emission spectra of the as-synthesized nanostructures of pure and doped CaF_2 samples, doped with different impurities at a concentration of 0.2 mole%.

to the nanoscale possibly could shift the emission bands to the lower wavelength, which might be due to a widening induced in the band gap of the material.

The PL emission spectra of Ag, Tb, and Cu doped samples shown in Figure 4 (curves (b), (d), and (e)) are almost similar to that of pure CaF₂ (curve (a)), but with a slight PL enhancement. The observed enhancement in case of Ag doped sample might be due to the increase in absorption and quantum yield. This absorption perhaps could be increased due to the surface plasmon resonance of Ag ions [40]. Ag dopants could perhaps be incorporated in metallic form [41]. It is also possible that some of these dopants could form small clusters; however, this is a preliminary speculation and further investigations are needed to prove the actual nature of Ag as a dopant in CaF₂ host.

The emission spectrum of Tb doped sample shows extra band at 544 nm. This band is the well-known emission of Tb³⁺ ion, which can be assigned to the ⁵D₄ → ⁷F₆ transition of Tb³⁺ ion [42]. In case of copper (Cu) ion it is possible that it might get incorporated in the host of LiF matrix in its 2+ form (Cu²⁺). This ion mostly shows its emission in the visible region. This ion was reported by several authors to have emission bands in the 400–500 region [43, 44]; therefore, the broad band at 370–550 nm might include the emission of Cu²⁺ and thus showed PL enhancement.

The PL emission spectra of Eu and Dy doped samples (Figure 4, curves (c) and (f)) show strong enhancement in intensity of the broad band at 370–550 nm, with the emergence of extra sharp emissions. The broad band at 370–550 nm has the highest PL intensity in Eu doped sample. This is in association with emergence of two bands located at 590 and 615 nm, which are the well-known emissions of Eu³⁺ ion [45]. Dy ion might get introduced into the host of CaF₂ matrix in its 3+ form (Dy³⁺) 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 [46], which are close to the emission region of pure CaF₂. The first emission of Dy³⁺ in CaF₂ matrix probably could enhance the PL emission of pure sample by superimposing these emissions.

Figure 5 shows the TL glow curves of CaF₂ nanostructures doped with different elements at a concentration of 0.2 mole% (curves (a), (b), (c), (d), and (e)). These samples were exposed to 1 kGy of ¹³⁷Cs gamma rays. The glow peaks of Eu, Cu, and Ag dopants (curves (a), (d), and (e)) are located at around 125°C with a variation in their relative TL intensity. Tb doped CaF₂ nanostructures (curve (c)) has stronger glow peak at lower temperature side, that is, around 95°C. The Dy doped sample (curve (b)) shows a broad TL glow curve with a prominent peak at around 165°C along with smaller one at 135°C. This curve has the highest TL intensity. This is a remarkable result to have a sensitive material with deeper traps, which is thermally stable with less fading. Moreover, Dy doped CaF₂ sample seems to have a good population of electron traps, making it a good candidate to be tested for its response to heavy ions used in radiotherapy like carbon ions. The nanostructure form of CaF₂ was tested by Zahedifar and Sadeghi [34, 47] and Zahedifar et al. [48] for its TL response

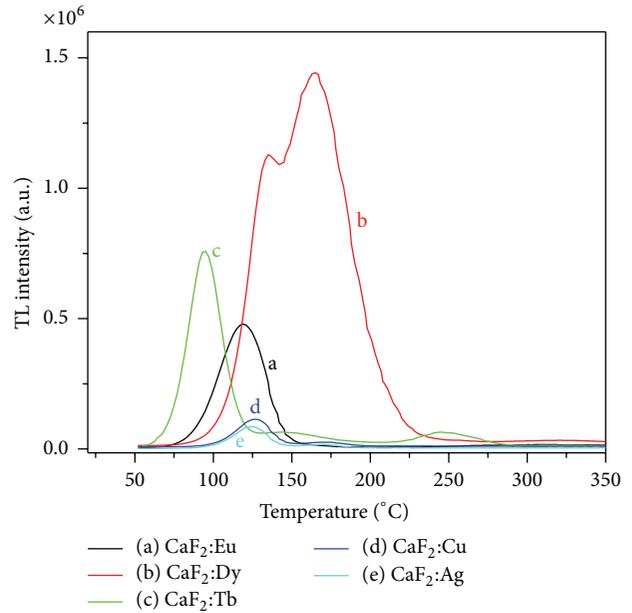


FIGURE 5: TL glow curves of CaF₂ nanostructures doped with different elements and exposed to 1 kGy of ¹³⁷Cs gamma rays.

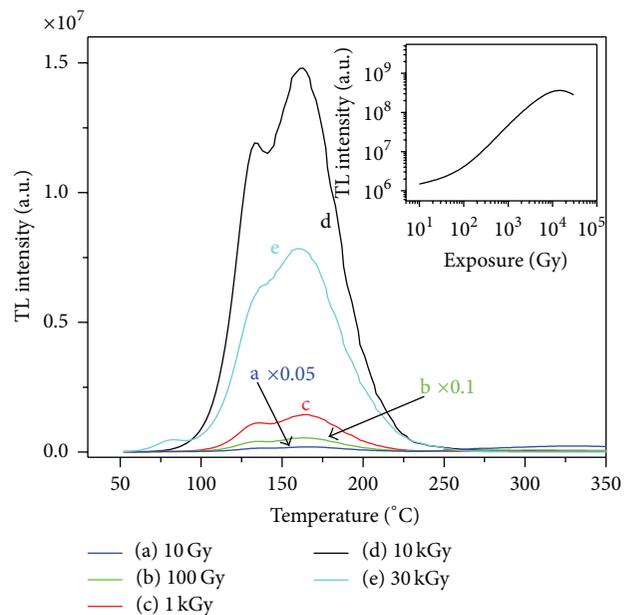


FIGURE 6: TL glow curves of CaF₂:Dy nanostructures exposed to different exposures of ¹³⁷Cs gamma rays. The figure in the inset is the corresponding TL response curve.

after doping with Tm, Ce. In their sample the permanent glow peak was observed at a relatively low temperature side, which is around 402 K (129°C). This makes CaF₂:Dy a superior due to its relatively high temperature glow peak. But it is quite useful to test the TL response of CaF₂:Dy nanostructures to different doses of gamma rays and observe the effect of different doses on the glow curve structure and peak position.

Figure 6 shows the TL glow curves of CaF₂:Dy nanostructures exposed to different exposures of ¹³⁷Cs gamma

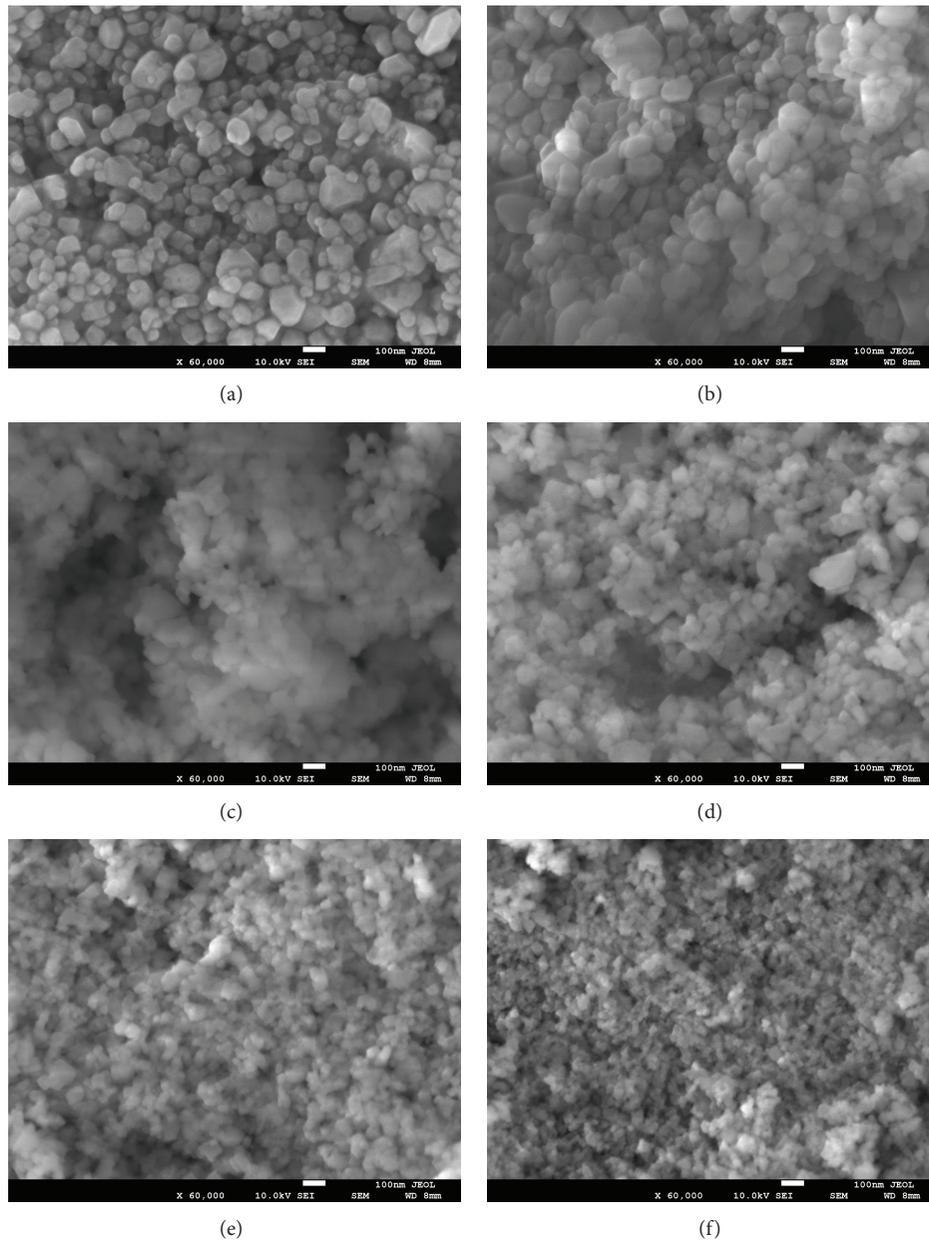


FIGURE 7: SEM images of (a) pure and Dy doped nanostructures of CaF_2 sample ((b) 0.05, (c) 0.1, (d) 0.2, (e) 1, and (f) 2 mole%).

rays in the range of 10 Gy–30 kGy. There is no significant change in the glow curve structure or peak position. The intensity of the glow peaks increased by increasing the dose in the range 10 Gy–10 kGy (curves (a), (b), (c), and (d)), but further exposure beyond this range results in decreasing the TL intensity of the glow peaks (curve (e)). Small TL glow peak emerged at around 70°C in case of 30 kGy exposure, which might be due to the saturation of electrons at the existing electron traps resulting in creating shallow traps.

The TL response curve of CaF_2 :Dy nanostructures to different exposures of ^{137}Cs gamma rays was also plotted and presented in the inset of Figure 6. It shows that below

100 Gy the curve is sublinear; then it is quite linear in the range of 100 Gy–10 kGy and finally saturates (even decreases) beyond this range. The linear response in a wide range is good indicator for CaF_2 :Dy nanocubes to be tested as a dosimeter for C ions. This wide response was explained earlier by Salah et al. [13, 49].

The effect of Dy concentration on the TL sensitivity of CaF_2 nanostructures has been studied. Different concentrations within the range 0.05–2 mole% were included in this study. The maximum TL sensitivity was found to be around 0.5 mole%. Strange result was also observed on the particle size of CaF_2 nanostructures by changing Dy concentration.

Figure 7 shows SEM images at the same magnifications for CaF_2 nanostructures doped with Dy at different concentrations from 0.05 to 2 mole% (images (a)–(f)). At low concentration (image (a)) the particle size is around 70–100 nm. This size was significantly reduced to around 20 nm by increasing concentration of Dy to 2 mole%. Similar results were also observed by Salah [50] on Tb doped CaSO_4 nanorods. The reason for that was attributed to the well incorporation of rare earth ions in the host that could limit the growth of these nanostructures; this means that these rare earths ions could act as controlling agents for size growth. The other impurities perhaps could not be incorporated well neither interstitially nor substitutionally. Rare earths like Dy, Tb, and Eu have ionic radii close to that of Ca, while those of Ag and Cu are different. Therefore, these rare earth ions perhaps could substitutionally be incorporated.

4. Conclusion

The TL and PL properties of CaF_2 nanostructures doped with Eu, Tb, Dy, Cu, and Ag were studied. Thermally stable nanocrystalline materials with a single phase in the 30–600°C temperature range could be produced. The PL emission spectrum of pure CaF_2 nanostructures has a broad band in the range of 370–550 nm. The doped samples showed similar spectra in addition to extra bands related to these impurities. The maximum PL intensity was observed in Eu doped sample. Dy doped one was observed to be the most TL sensitive with a linear response curve in the 100 Gy–10 kGy range. This impurity could also play a role as a controlling agent for particle size growth. These results showed that Dy is a proper activator in the host of CaF_2 nanostructures, providing a highly sensitive dosimeter in a wide range of exposures that might be suitable for measurements of heavy doses.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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