



Thermoluminescence and photoluminescence properties of NaCl:Mn, NaCl:Cu nano-particles produced using co-precipitation and sono-chemistry methods



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ABSTRACT

The NaCl: Cu and NaCl: Mn nanoparticles (NPs) were produced by co-precipitation and sono-chemistry methods and their thermoluminescence (TL) and photoluminescence (PL) properties were studied. By decreasing the particles size a considerable increase in sensitivity of the samples to high dose gamma radiation was observed. The NPs produced by sono-chemistry method have smaller size, homogeneous structure, more sensitivity to high gamma radiation and less fading than of those produced by co-precipitation method.

1. Introduction

Using the sound waves in chemical solutions is common to synthesis nanoparticles (NPs). Loomis [1] studied the chemical effects of ultrasonic waves and extensive experimental studies in this field have been started since 1980 when the cost effective ultrasonic devices came to the market. A major advantage of ultrasonic radiation is that the shape and size of the nanoparticles can be adjusted by varying the operating parameters [2] by which we can initiate various chemical reactions by generating free chemical ions (radicals); accelerate chemical reactions by facilitating the mixing of reactants; enhance polymerization and depolymerization reactions by temporarily dispersing.

The synthesis of the nano structures using the ultrasonic waves was reported by Suslick [3,4] group for the first time in 1991. They synthesized the amorphous Fe nanoparticles with dimension of 10–20 nm by irradiating Fe(Co)₅ by the ultrasonic waves. They also used this method to synthesis inorganic NPs with the Carbonyl structure of other metals like Mo(CO)₆Co(CO)₃NO and W(CO)₆ [5–8]. Since then Sono-chemistry is known as one of the efficient synthesis methods in production of NPs.

The effect of ultrasonic waves are used usually in both thermal and hydro-co-precipitation processes in which clustering and overgrowth are possibly occurring. The synthesis of NPs [9] by co-precipitation method is carried out in four steps:

- Preparation of the supersaturated solution
- The core formation
- The core growth and
- The accumulation of particles.

A uniform and homogeneous blending of the solution can help to make particles with a more uniform size. One can use the ultrasonic waves, microwaves or neutral gas stream [10] instead of mechanical mixtures. The effects of ultrasonic wave on alkali halide crystals (NaCl and KCl) have been studied recently [11,12].

Alkali halide NaCl is one of the most abundant phosphorus which is suitable for archaeological dating due to its high sensitivity to ionizing radiation [13]. In addition when we dope the NaCl crystals with certain impurities, it shows interesting TL as well [14]. Sodium chloride with different impurities like Ce, Mn, Cu, Co and Dy are manufactured and their TL and PL properties are studied [14–18].

The phenomenon of thermoluminescence can be described as follows. A solid sample, usually an insulator material, is excited, characteristically by ionizing radiation at certain low temperature. Yet another version is when it is a naturally occurring material irradiated by the radiation field in its natural surroundings. At the end of this stage, the sample is placed in an appropriate oven. This period of heating is also called “read out” stage. At this stage, the temperature of the oven is raised gradually, typically at a constant heating rate and light emission as a function of temperature is recorded

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using a light sensitive detector such as a photomultiplier. This emitted light as a function of temperature yields the so-called thermoluminescence glow curve [19].

It is well known that the physical properties of materials with nano size are different from those of bulk equivalents [20]. The luminescence property is highly dependent on the size of the material.

Recently, Alkali halides specially NaCl are studied because they have potential to be used as a dosimeter [21]. The early works performed on NaCl are summarized by McKeever [19].

The kinetic parameters of the glow curve play an important role in the behavior of phosphorus dosimeters irradiated with ionizing radiation. The crystal of NaCl doped with Ca and Mn impurities is characterized as a dosimeter recently. The effective atomic number of this dosimeter is 15.7 which is close to that of the human body bone. Therefore, one can use this crystal to measure the absorbed dose in human body in low temperature [16].

The NPs of pure NaCl doped with Mn and Cu are synthesized and their TL and PL properties are studied in this work. Both Co-Precipitation and Sono-chemistry methods are used in synthesis of the NPs.

2. Synthesis of NaCl:Mn and NaCl:Cu NPs

2.1. Co-precipitation method

The process is started by solving 0.3g pure Na in 10 ml of ethanol in stirrer. While the solution is blended by a magnetic-blender, 2.08g diethylmalonate, 10 ml toluene and finally 2.58g acetyl chloride are added to the solution. All materials are the pure one from MERCK company. The result of the reaction is formation of a white precipitate that is separated from solution by a centrifuge and then washed few times with ethanol. Finally, the obtained sample is dried in an oven at a temperature of 100 °C to have the NaCl NPs. The proper amount of manganese chloride and copper nitrate are added to the solution of ethanol and Na to dope the desire impurities.

2.2. Sono-chemistry method

In this method we used the same materials and procedure as mentioned in the co-precipitation method except that during the synthesis process which takes about 5 min, the solution is irradiated with an ultrasonic wave with a frequency of 20 kHz to prevent the rapid growth of the Nps.

3. Analysis of NaCl:Mn and NaCl:Cu NPs

3.1. The scanning electron microscope (SEM) image and X-ray diffraction pattern (XRD)

The NPs of NaCl: Mn and NaCl: Cu can be produced with desired size and structure by exact controlling reaction parameters and optimizing them. The SEM image, size distribution and the XRD pattern of produced NPs are shown in Figs. 1–3, respectively....

The obtained results confirm the formation of NaCl crystals which compiles perfectly with reference card number 77-2064. The size of the NPs can be estimated by the Scherrer equation [22].

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where D is the average diameter of the nanoparticles, λ is the wavelength of Cu K_α (0.1543 nm) radiation, β (in radians) is the full width at half maximum (FWHM), and θ is the Bragg angle. The average particle size was found to be 92 nm.

3.2. The PL properties of NPs

The emission spectrum and excitation wavelength for NPs were obtained in this work. The emission spectrum of the NaCl: Cu produced by co-precipitation method which is shown in Fig. 4a has a peak at wavelength of 332.5 nm. The spectrum of produced Nps using ultrasonic wave shows a peak at a wavelength of 330.0 nm. The excitation wavelength of this Nps is 275 nm. The emission spectrum of bulk NaCl: Cu crystal has excitation wavelength of 255 nm and emission wavelength of 350 nm that corresponds to an electron transition between 4 s and 3d and 3d10 states [23].

Fig. 4b illustrates the emission of the NaCl: Mn NPs produced by co-precipitation and sono-chemistry methods. The excitation wavelength of this NPs is 250 nm and the emission spectrum shows peaks at wavelengths of 610 and 605 nm for the Co-Precipitation and Sono-chemistry method, respectively. The PL spectrum of bulk NaCl: Mn crystal excitation at wavelength of 320 nm is peaked at wavelength of 610 nm which is attributed to electron transition from 4 s state to 3d state of Mn impurity [16]. Shifting the emission wavelength to the lower value is an indication of decrease in the particles size which is observed in sono-chemistry method [24].

3.3. The TL glow curve and dose response of NaCl:Cu and NaCl:Mn NPs

The TL intensity of the NaCl: Cu and NaCl: Mn samples produced by two different methods are shown in Fig. 5(a) and (b), respectively. A computerized glow curve deconvolution (CGCD) technique was used to obtain the trapping parameters of overlapping glow peaks. The computer program has been produced in our laboratory using the Levenberg Marquart algorithm based on non-linear least square method. Thermoluminescence general order glow curve deconvolution function (Eqs. (2) and (3)) was used in our program for curve fitting [25].

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

$$s = \frac{\beta E}{kT_m^2 Z_m} \exp\left(\frac{E}{kT_m}\right) \quad (3)$$

with $\Delta = \frac{2kT}{E}$, $\Delta_m = \frac{2kT_m}{E}$ and $Z_m = 1 + (b-1)\Delta_m$ and where I is the glow-peak intensity, E (eV) the activation energy of the trapped carriers, k (eVK⁻¹) the Boltzmann constant, T (K) the absolute temperature, β the heating rate and b the kinetic order..

The used function in terms of I_m , T_m , b , E instead of s , n_0 , b , E (n_0 , cm⁻³ is the initial population of trapping states) has advantage that I_m and T_m can easily be estimated from the shape of the glow curve as initial values in glow curve deconvolution process. Also the used function is more general than the deconvolution functions for limiting cases of first and second orders of kinetics since intermediate cases in which $b \approx 1$ or 2 can be dealt with and it smoothly reduces to the first and second orders when $b \approx 1$ and $b \approx 2$, respectively. The fitting quality is checked by FOM [26] which is presented in Eq. (4)

$$FOM = \sum_{j_f}^{j_l} \frac{100|y_i - y(x_i)|}{A} \quad (4)$$

in which j_f and j_l are the numbers of the first and last temperature interval ΔT used for curve fitting, Y_i is the intensity in the i th interval obtained from experiment and $Y(x_i)$ the intensity expected.

Comparison between the obtained results shows that particles with smaller size have a much higher sensitivity to the high dose gamma radiation. The TL intensity of NaCl: Mn and NaCl: Cu NPs produced by

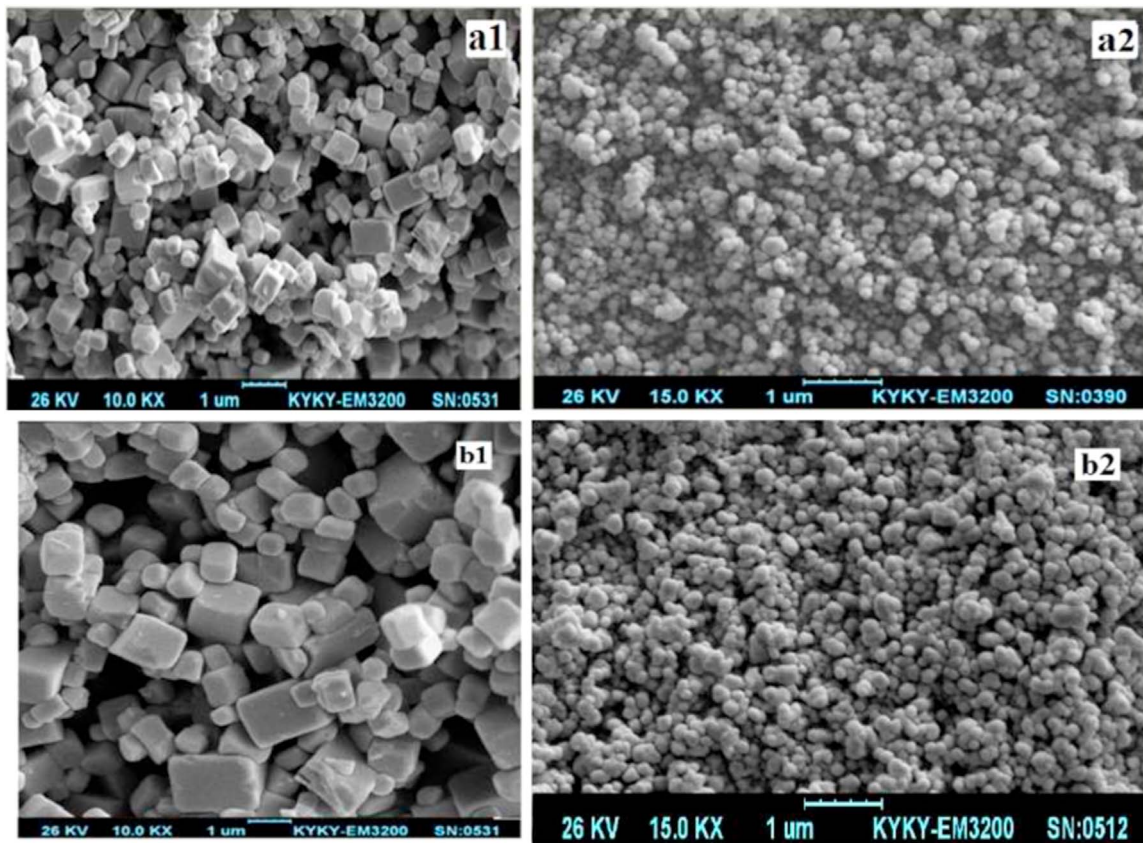


Fig. 1. The SEM image of NaCl:Mn and NaCl:Cu produced by co-precipitation method (sono-chemistry method) are indicated as a1 and b1 (a2,b2).

sono-chemistry method is almost two and three time higher than that of NPs produced by co-precipitation method. The size of NaCl: Mn NPs produced by sono-chemistry method is almost five time smaller than the NPs produced by co-precipitation method.

Figs. 6 and 7 respectively represent the component TL glow peaks contained in complex glow curves of produced NaCl: Cu and NaCl: Mn NPs which have been deconvolved using the computed glow curve deconvolution program. The produced samples were exposed using a ⁶⁰Co source with doses from 50 Gy up to 10 kGy...

Fig. 8 shows the TL dose response of NaCl: Cu and NaCl: Mn samples as a function of exposed dose..

3.4. The effect of thermal treatment on TL sensitivity of the samples

In order to empty the deep traps and also obtain more regular crystals, the samples were annealed. The proper temperature and annealing duration for each TL material was 8gft obtained experimen-

tally. The produced samples were annealed for 15 min in different temperatures and then are cooled by putting over an Al plate. The TL intensity of non-annealed and annealed samples of NaCl: Cu in different temperatures is shown in the left panel of Fig. 9. Shown in the right panel are the same results for NaCl: Mn except that the solid glow curve in the right panel belongs to not annealed and not irradiated sample. The population of trapping states for this sample is due to the excitations by chemical reactions during the preparation procedure. The minimum annealing temperature of 450 °C with a duration of 15 min was required to empty these traps. As is seen in Fig. 9b, annealing at 500 °C has not affected the TL sensitivity. Therefore, the optimum annealing regime for NaCl: Mn sample was obtained as 450 °C for 15 min.

3.5. Effect of impurity concentration on TL sensitivity of samples

It is known that aliovalent impurities enter an alkali halide lattice

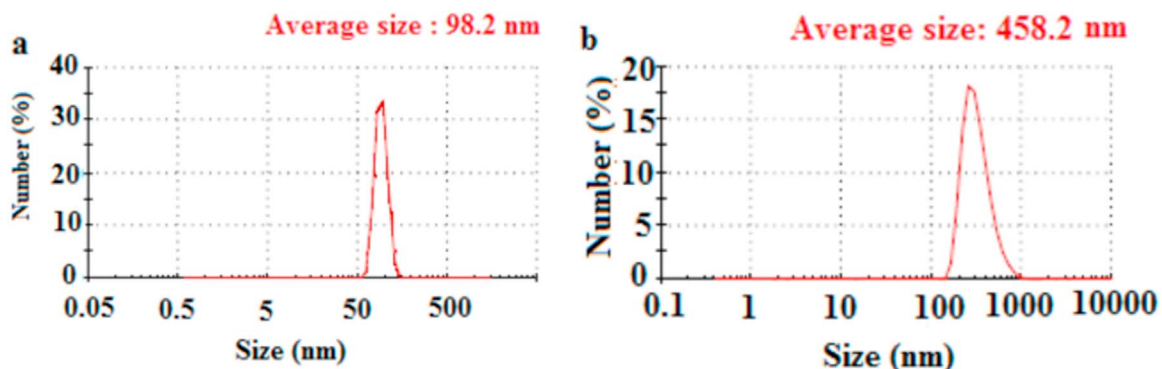


Fig. 2. Size distribution of NaCl NPs synthesized by a) sono-chemistry b) Co-Precipitation.

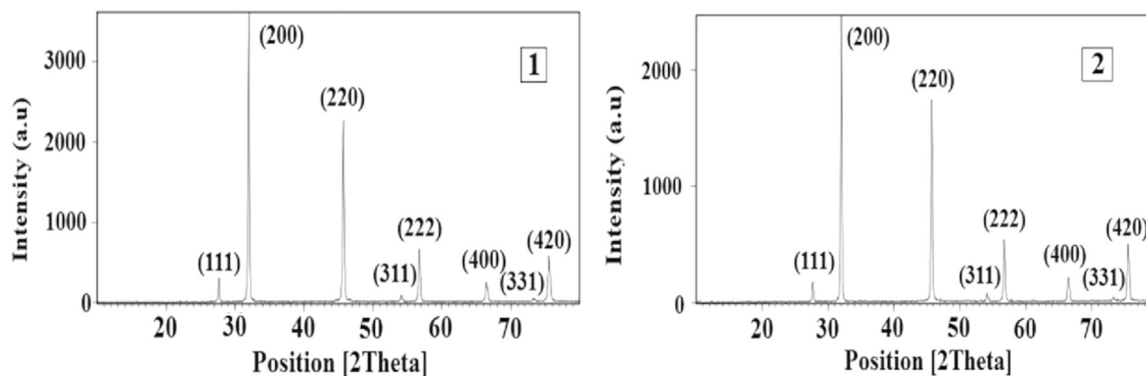


Fig. 3. The XRD pattern of NaCl:Mn and NaCl:Cu produced by co-precipitation method (sono-chemistry method) are indicated as 1 and (2).

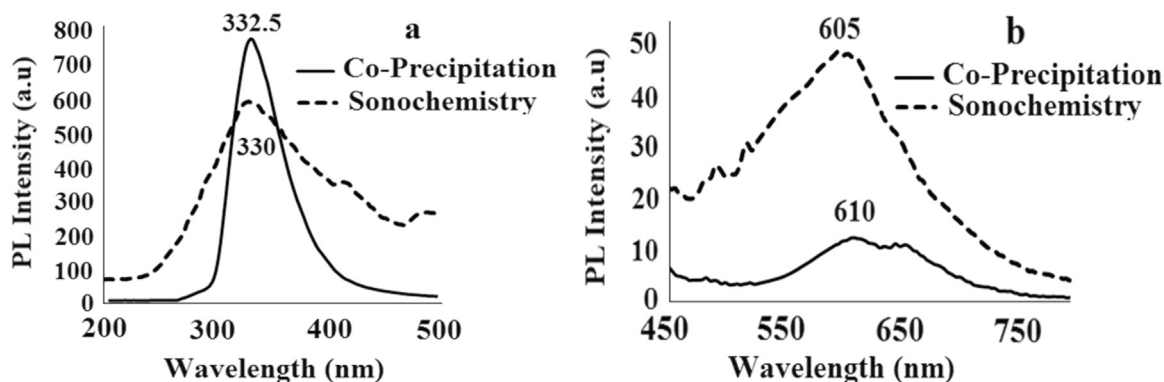


Fig. 4. The emission spectrum of the produced samples by co-precipitation and sono-chemistry methods, NaCl:Cu (a) and NaCl:Mn (b).

substitutionally and require the presence of host ion vacancies of the appropriate sign in order to maintain charge neutrality [21]. The inserted impurity or complexes of impurity and vacancies are responsible for trapping of charge carriers produced upon irradiating the sample with ionizing radiation. Therefore the kind and amount of impurities has crucial role on TL sensitivity. The effect of impurity on TL sensitivity of samples is shown in Fig. 10. Results show that the optimum value of the impurity concentration which give rise to the maximum sensitivity is 0.1% and 0.3% mol for NaCl: Cu and NaCl: Mn NPs produced by co-precipitation and is 0.3% and 0.1% mol for the NPs produced by sono-chemistry..

3.6. Trapping parameters and fading characteristics of the samples

As pointed out earlier, the TL glow curve deconvolution function presented in Eqs. (2) and (3) was used to deconvolve the complex TL glow curves and obtain the trapping parameters of the component glow peaks. From 4 adjustable parameters I_m , T_m , E and b in Eq. (2), I_m

and T_m can easily be estimated from the TL glow curve as input parameters and the activation E and kinetic order b are obtained as a result of fitting process. The results are shown in Table 1A and B respectively for trapping parameters of NaCl: Cu and NaCl: Mn NPS. The uncertainties in E and b are results of fitting processes conducted for TL glow curves obtained from seven samples prepared at the same conditions. It is known that The activation energy and the frequency factors are the main parameters determining the stability of the TL glow peak. Having the activation energy, Kinetic order and the peak maximum temperature of a TL glow peak, the frequency factor s was calculated from Eq. (3).

The fading of the NaCl: Cu and NaCl: Mn samples produced by co-precipitation and sono-chemistry methods during a period of 60 days is shown in left and right panels a and b of Fig. 11. The fading of the NaCl: Cu NPs produced by co-Precipitation is 80% and the one made from sono-chemistry is 78% that is negligible difference. In case of NaCl: Mn the corresponding values are 74.5% and 39%. Therefore, the NaCl: Mn NPs produced by sono-chemistry method show the more

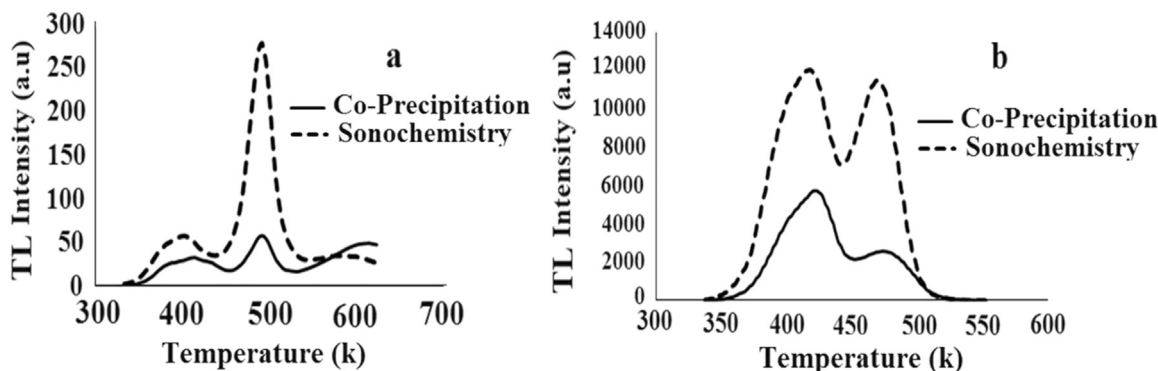


Fig. 5. The TL intensity of NaCl:Cu (a) and NaCl:Mn (b) samples produced by two different methods.

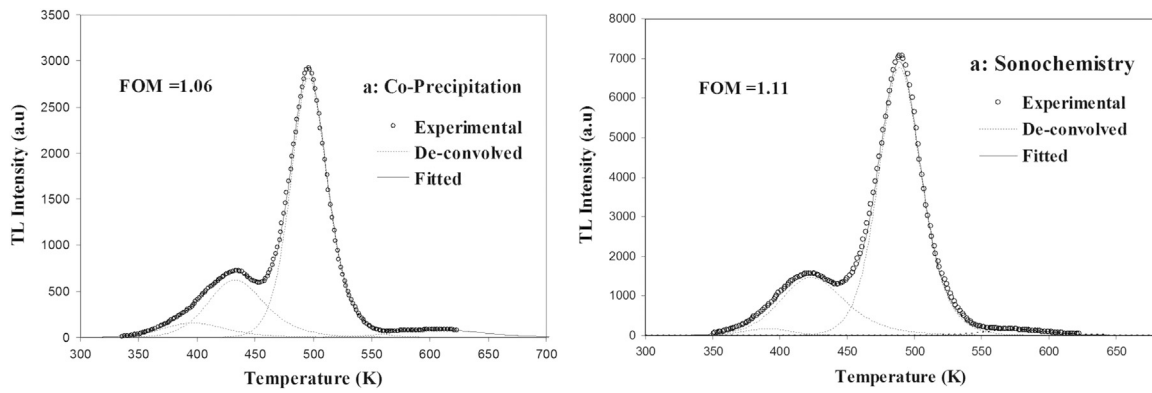


Fig. 6. The fitting result of the TL dose response for NaCl:Cu produced by two methods.

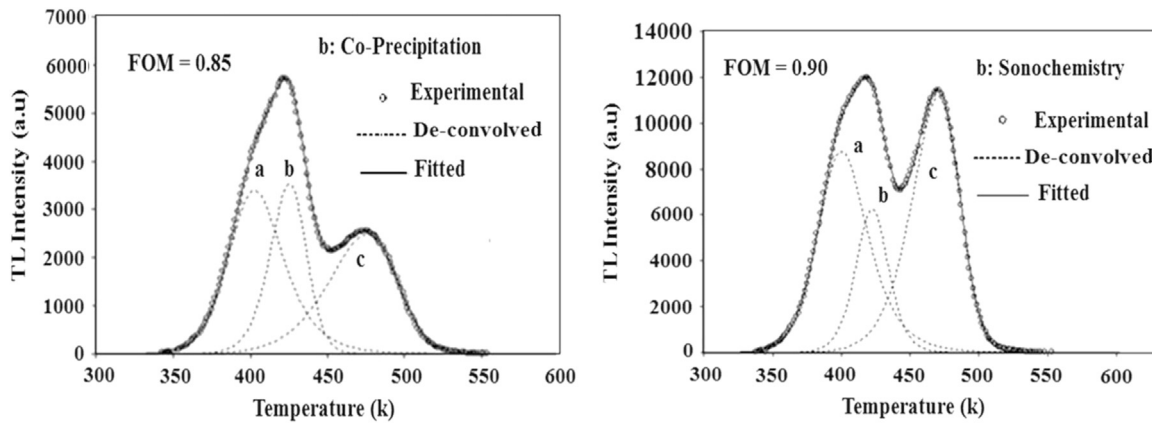


Fig. 7. The fitting result of the TL dose response for NaCl:Mn produced by two methods.

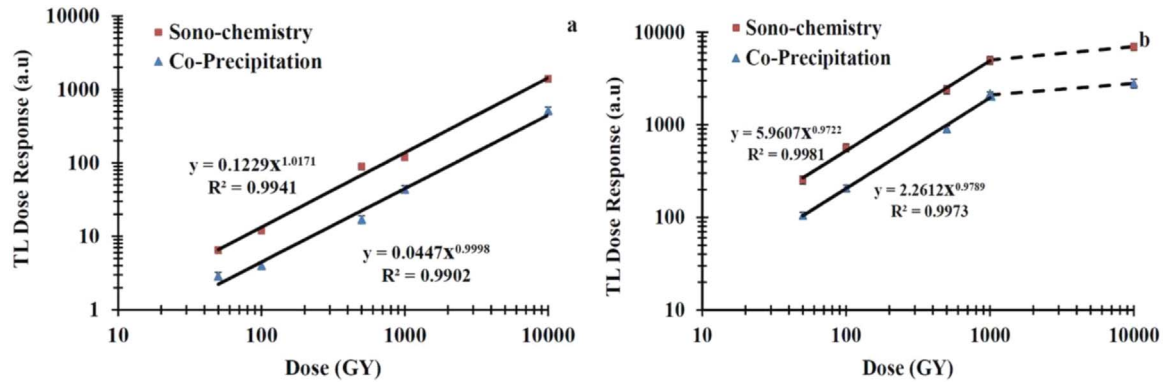


Fig. 8. The TL dose response of samples as a function of exposed dose. a) NaCl:Cu b) NaCl:Mn.

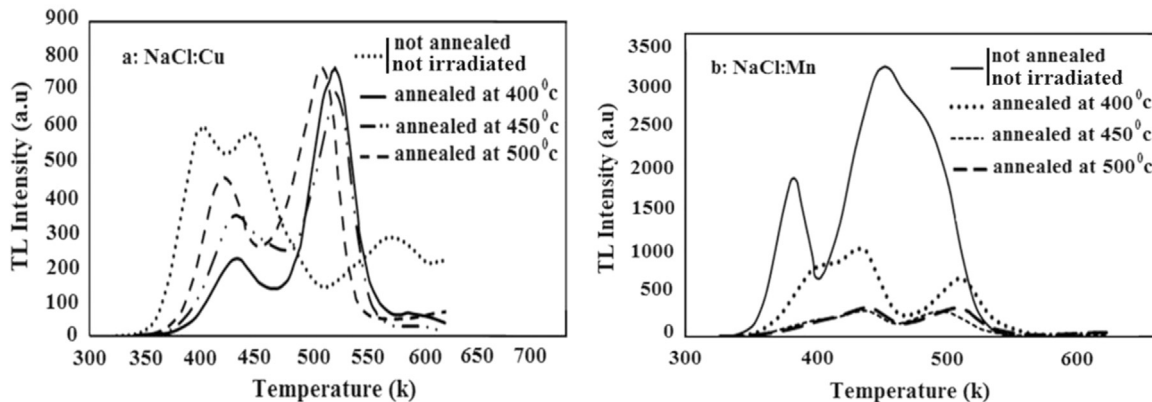


Fig. 9. The TL intensity of non-annealed and annealed samples in different temperatures are shown.

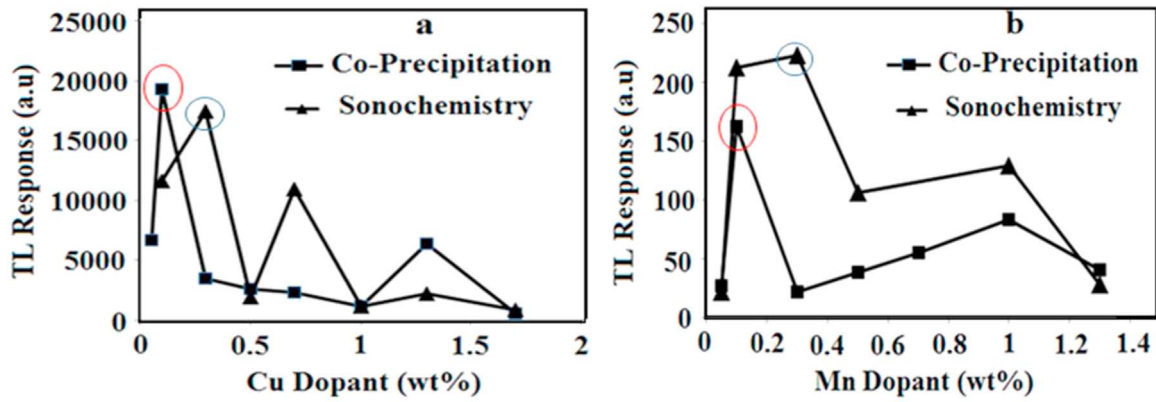


Fig. 10. The effect of impurity concentration on TL sensitivity of a:NaCl:Cu,b:NaCl:Mn.

Table 1
The estimated kinetic parameters of the NaCl: Cu (A) and NaCl: Mn(B) samples.

Peak	Synthesis method	b	Δb (%)	E (eV)	ΔE (%)	T_m (K)	ΔT_m (%)	$s(s^{-1})$
A								
a	Co-Precipitation	1.85	1.25	1.2	3.24	396	0.5	1.58E+14
a	Sono-chemistry	1.71	2.58	1.2	2.57	391	0.4	2.50E+14
b	Co-Precipitation	1.98	2.52	1.02	0.97	432	0.5	4.70E+10
b	Sono-chemistry	2.05	3.68	1.10	5.29	423	0.4	8.52E+11
c	Co-Precipitation	1.99	6.02	1.54	4.23	496	0.5	3.06E+14
c	Sono-chemistry	1.97	2.99	1.61	4.06	489	0.4	2.28E+15
d	Co-Precipitation	1.93	8.85	1.60	5.02	605	0.5	1.01E+12
d	Sono-chemistry	1.99	3.94	1.85	3.44	575	0.4	1.01E+15
B								
a	Co-Precipitation	2.12	4.23	1.18	6.22	403	0.5	4.52E+13
a	Sono-chemistry	2.10	4.01	1.14	7.06	400	0.4	1.79E+13
b	Co-Precipitation	1.45	5.47	1.63	5.21	425	0.5	2.19E+18
b	Sono-chemistry	1.76	4.25	1.43	4.14	423	0.4	9.74E+15
c	Co-Precipitation	1.14	1.08	1.75	3.33	475	0.5	3.30E+17
c	Sono-chemistry	1.21	2.45	1.30	1.87	470	0.4	5.87E+12

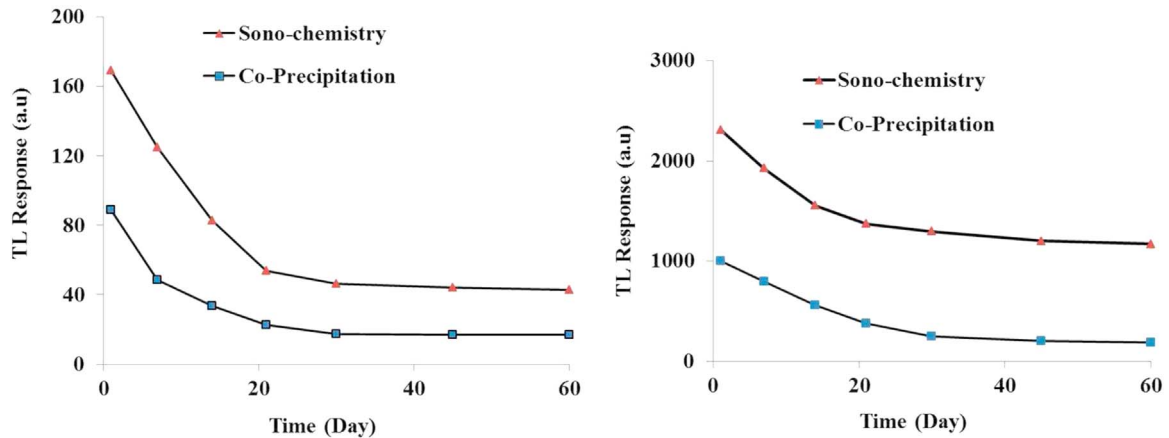


Fig. 11. The fading of the samples produced by co-precipitation and Sono-chemistry methods during a period of 60 days. A) NaCl:Cu b)NaCl:Mn.

stability during a period of 60 days..

Considering the high fading of the produced NPs, it is constructive to evaluate the half-lives of different glow peaks contained in TL glow curve of NaCl: Cu and NaCl: Mn NPs and compare them with the above results for fading. The intensity for a single TL glow peak in general order of kinetics is

$$I = -\frac{dn}{dt} = sn^b \exp(-E/kT) \quad (5)$$

In which I is the TL intensity, $n(\text{cm}^{-3})$, the population of trapping state, $E(\text{eV})$, the activation energy, b , the order of kinetics, k the Boltzmann's

constant and $T(K)$, the temperature. Integrating the above equation for a constant temperature yields

$$n = [n_0^{b-1} - s(b-1)\exp(-E/kT)t]^{-\frac{1}{b-1}} \quad (6)$$

Where n is the initial concentration of trapping state at $t=0$. The above equation gives the variation of the trapped charge carriers (n) with time for a constant temperature. By equating $n = n/2$ in the above equation, the half life, τ is obtained as:

$$\tau = \frac{2^{b-1} - 1}{(b-1)s} \exp(E/kT) \quad (7)$$

For given values of s , b and E , the half life for a single glow peak at room temperature ($T = 300$ K) can be evaluated. Using the above equation, the values of 9, 11444 and 10^4 days obtained for half lives of peaks 1, 2 and 3 of NaCl: Mn NPs prepared by sono-chemistry method, respectively. The corresponding values for the NaCl: Mn NPs produced by co-precipitation method are 17, 10^4 and 6×10^6 . The above evaluated half lives are in accordance with the experimental results for fading of NaCl: Mn NPs. The calculated half lives for peaks 1, 2 and 3 of NaCl: Cu NPs prepared by sono-chemistry method are 6, 41 and 4×10^6 days in that order. The corresponding values for the NaCl: Cu NPs produced by co-precipitation method are 10, 33 and 3×10^6 . It is apparent that the calculated half lives for NaCl: Mn NPs predict lower fading at room temperature. The high fading of NaCl: Cu NPs can be explained by significant tunneling of charge carriers from the trapping states responsible for the more stable peak 3 to lower temperature peaks 1 and 2. At the same time fading of the samples were tested for three months. In fact after two months no significant change in the fading characteristics of two produced NPs were observed.

4. Discussion and conclusion

The pure NPs of NaCl doped with Mn and Cu as impurities, are synthesized using co-precipitation and sono-chemistry methods and their TL and PL properties are studied. The obtained results are summarized in the following items.

- The x-ray pattern of the synthesized NPs confirms that the formed structure produced by co-precipitation method and also by sono-chemistry method is NaCl crystal. According to the SEM image of the samples one can see that the NPs produced by sono-chemistry method have smaller size and have a spherical shape whereas those produced by co-precipitation method have bigger size and a cubic shape. The difference between the shapes of the samples produced by two methods is attributed to the effects of ultrasonic wave.
- Decreasing the particles size causes considerable increase in sensitivity of the samples to high doses of gamma radiation. The TL sensitivity of the NaCl: Cu and NaCl: Mn NPs produced by sono-chemistry method is almost three and two times bigger than the NPs produced by co-precipitation method.
- The produced samples show a linear response to the exposed dose of 50 Gy up to 10 kGy. The NaCl: Mn NPs produced by sono-chemistry method shows less fading which indicates the stability of TL signal of this sample and finally
- The NPs produced by sono-chemistry method have smaller size, more homogeneous structure, higher sensitivity to the high gamma radiation and less fading than of those produced by co-precipitation method. Therefore, the sono-chemistry method is recommended for production of NaCl nanoparticles. Fig. 9
- shows impressive results. It shows how important is the type of the impurity. In the case of Cu-impurity, a thermally stable material is obtained whereas the Mn-impurity gives an extremely thermal sensitive and unstable material. In other words, the may be more preferable than the. The significant reduction of TL sensitivity of NaCl: Cu NPs can be attributed to the destruction of TL trapping states mediated with Cu impurity while the complexes responsible

for TL trapping states of NaCl: Mn NPs remain stable with increasing the annealing temperature.

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