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## Abstract

Objectives: <sup>[0]</sup>▶ The purpose of this research is to synthesize the Yttrium doped PEG-coated ZnO nanostructures and characterize them especially as thermoluminescence material for gamma dosimetry. <sup>[0]</sup>▶ The dosimetry characteristics were evaluated by irradiating the sample with gamma radiation with different doses (10 and 100 Gray) at a temperature range of 0-400°C.

Methods: Uncoated undoped ZnO NPs and PEG-coated ZnO Nanoparticles (NPs) with 0, 5, 7, and 10 at% Yttrium (Y) doping concentration were synthesized via hydrothermal technique. Synthesized nanoparticles were examined through various characterization techniques (XRD, SEM, EDS, UV, Tyndall test and TLD) for their morphology, PEG-coating confirmation and to evaluate their Thermoluminescence properties

Results: XRD analysis confirmed the wurtzite hexagonal structure and doping of Yttrium into ZnO. SEM analysis showed PEG-coated ZnO has well-rounded nanoplates 100nm confirming the successful growth inhibition of PEG working as surfactant. The particle size was found to grows with increasing Y concentration. <sup>[30]</sup>▶ UV visible results reveal an increase in the optical band gap of ZnO with Yttrium doping. With doping, TL response has enhanced significantly, and glow peak shifted towards higher temperature. <sup>[0]</sup>▶ TL analysis showed two broad peaks following the second-order of kinetics and five traps with activation energies 1.25, 0.87, 0.75, 0.82, 1.99 eV, present in the Y doped ZnO.

Conclusions: Y doped ZnO is a suitable candidate for high-dose thermoluminescence dosimetry.

Keywords: TLD; Hydrothermal; Doped; Gamma Dosimetry, Thermoluminescence

Abbreviations: TLD, Thermoluminescence dosimeter; UV, Ultraviolet; FOK, First-order kinetics; PEG, Polyethylene Glycol; SOK, Second-order kinetic; PL, Photoluminescence

## Introduction

The quest for an efficient, sensitive, and versatile phosphor is never-ending owing to their ever-emerging use in optical dating, high power LEDs, fluid temperature sensors (Huntley et al., 1985; Chung et al., 2019; Abram et al., 2018), etc. This quest has produced many attractive phosphors among which ZnO has been the focus of researchers worldwide because of its facile synthesis, high binding energy (60 meV), broader bandgap (~ 3.35 eV) (Kumaresan et al., 2017'a'; Hjiri et al., 2017), potential uses as sensors, fast scintillators, transparent conductive electrodes, particle detectors (Grigorgeva et al., 2015; Liu et al., 2013; Neal et al., 2009). But pure ZnO has little to low potential for the above-mentioned application in its intrinsic form, due to its weak optical characteristics (Kumaresan et al., 2017), doping can, however, enhance ZnO potential for the above-mentioned applications (Kumaresan et al., 2017'b'). Ü. Özgür et al. showed extensive studies for assessing chemical, optical, physical, electrical, thermal, and mechanical characteristics of ZnO. **Recently, doped ZnO NPs has attracted researchers attention as a thermoluminescent material and has been evaluated for its thermoluminescence by doping with Silver, Magnesium Gadolinium etc.**(Abraheem et al., 2021; Isik and Gasanly, 2019; Thabit et al., 2021a, 2021b). The luminescence of phosphor can be shifted from the UV region to the lower region by employing doping techniques (Dhar et al., 2005).

<sup>[16]</sup>▶ ZnO **nanomaterials have been fabricated by using innovative methods to get different morphology and dimensions (Zhang et al., 2009).**<sup>[10]</sup>▶ The yield of nanostructures in large amounts is a challenge and hence **there is a need for the development of new technologies for material fabrication (Nickel et al., 2006).**<sup>[10]</sup>▶ **Chemical methods for the synthesis of nano-materials are found to be superior in terms of cost and control of synthesis parameters.** ZnO nanoparticles have enormous applications owing to their quantum confinement effect, enhanced surface area, and surface energy (Sun et al., 2006). Recently, researchers have been keen to explore the quantum size effects of doping on material physical properties. Several studies have shown the effects of doping of different metals in ZnO and its enhanced properties for gas sensing, optoelectronics and photocatalysis, dielectric and ferroelectric properties (Goel et al., 2018, Bharat et al., 2019). Dopants are used to modulate material properties in desirable ways, due to which these play an important role in semiconductor devices.<sup>[16]</sup>▶ **Among doping materials, trivalent transition elements as dopants have attracted a lot of**

attention due to the use of end products in major fields including solar applications and thermoluminescence properties. (Liu et al., 2007).

Optical energy bandgaps, conductivity, and band structure influence by Y doping and its potential use as a gas sensor have been studied (not making sense, needs rephrasing). Yogamalar et al. have prepared ZnO: Y through hydrothermal route and discussed bandgap narrowing in materials employing different dopant concentrations using UV-vis. Morphological changes observed through SEM and TEM were related to dopant concentration in their study. Zheng, J. H., et al. prepared Yttrium-doped ZnO nanoparticles via a sol-gel method that exhibited increased photoluminescence and enhanced UV emission. A bunch of nano-geometries have been developed by using Y as a dopant in ZnO with versatile applications of each shape like Nanosheets as piezo-electric devices (Sinha et al., 2018), nanorods as a catalyst for acetone (Yu et al., 2013), and nanospheres as gas sensors (Guo et al., 2013).

Detectors working on Thermoluminescence principal are termed Thermoluminescence Dosimeters(TLD). Dosimeters are employed in detecting dose for food sterilization, preservation, material processing, and personnel (Kalpana et al., 2017; Sanyal et al., 2019). TLDs are preferred as they can act as human tissue equivalent which makes them perfect for dose measurement purposes (Dubey et al., 2014). Common TLD materials made up of ceramics, crystals and phosphors have the problem of fast fading, saturation, moisture attack, difficulty in annealing, and opaque nature (Dubey et al., 2014; Swamy et al., 2014; Saidu et al., 2019). Thabit et al. used a co-doping technique employing silver as co-dopant in ZnO: Y nanoparticles obtaining nano-flowers and nano-rod geometry showing some promising results to use it as a TLD dosimeter.

Based on these studies, this study aims at a systematic investigation of Y doping in PEG-coated ZnO and its effect on morphology, crystal structure, Special focus is given to the potential of these nanostructures as TLD materials for measuring gamma irradiation dose. Hydrothermal synthesis was chosen in this study owing to its better compositional and morphological control (Yoshimura et al., 1999). The results suggest that PEG coating and Y doping both affect profoundly the morphology and luminescence characteristics of the nanostructure. These promising results suggest that ZnO: Y<sup>[18]</sup> can be a good dosimeter for high-dose applications like irradiation of foods and seeds.

## Materials and Methods

Zinc sulfate heptahydrate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) Sigma Aldrich 99.99%, Sodium Hydroxide ( $\text{NaOH}$ ) 99.99%, Yttrium Oxide ( $\text{Y}_2\text{O}_3$ ), commercially available Nitric Acid ( $\text{HNO}_3$ ), PEG were used for the preparation of pure and doped material. Pure and Y doped ZnO materials (of 0, 5, 7, 10 mol%) were prepared through the hydrothermal method (Gupta et al., 2014).

For a typical reaction 60ml of 0.5M  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  aqueous solution was prepared using de-ionized water, pH of the solution was maintained using  $\text{NaOH}$ . The solution was then moved to the hydrothermal autoclave for the reaction at  $150^\circ\text{C}$  for 8 hours. After cooling, particles were collected and washed with deionized water. NPs were obtained by drying them in an oven at  $80\text{-}90^\circ\text{C}$  for 12 hours, the product obtained was converted to a fine powder using mortar and pestle. For PEG-coated products, the same procedure was repeated with equal volumes of  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  and PEG. In the synthesis of yttrium (Y) doped ZnO NPs above procedure was repeated with  $\text{Y}(\text{NO}_3)_3$  as an additional precursor. The summary of synthesis is provided in Table 1 with suitable Labeling for each sample. Molarity was calculated by the following equation:

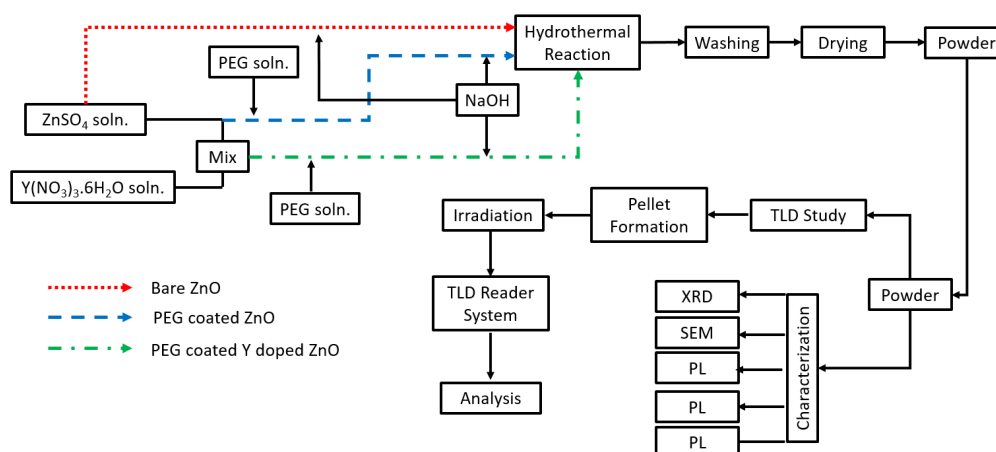
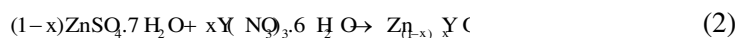


Figure 1: Schematic diagram of work being done.

Table 1. Summary of synthesis with different doping concentrations.

		Conc. (M)		Quantity (ml)			Required Weight (g)		
		ZnSO <sub>4</sub> ·7H <sub>2</sub> O	Y(NO <sub>3</sub> ) <sub>3</sub> ·6H <sub>2</sub> O	ZnSO <sub>4</sub> ·7H <sub>2</sub> O	PEG	Y(NO <sub>3</sub> ) <sub>3</sub> ·6H <sub>2</sub> O	ZnSO <sub>4</sub> ·7H <sub>2</sub> O	PEG	Y(NO <sub>3</sub> ) <sub>3</sub> ·6H <sub>2</sub> O
		0.5	0	30	30	0	4.31	3	0
		0.5	0	30	30	0	4.31	3	0
		0.475	0.025	25	25	10	3.41	2.5	0.0957
		0.465	0.035	25	25	10	3.34	2.5	0.134
		0.45	0.05	25	25	10	3.23	2.5	0.192

### Characterization Techniques

Structural characterization of the prepared samples were done using X-ray diffraction (Philips X'Pert Pro MPD) with Cu K $\alpha$  radiation ( $\lambda=1.54 \text{ \AA}$ ). EDS was performed for qualitative analysis, UV-Vis analysis for bandgaps, and Tyndall test. The morphological studies were done using SEM (FESEM, MAIA-3, TESEAN) with an electron beam of 10kV. TL studies were done for prepared pellets using the TL RA94 reader available at the lab facility. Spent FUEL PARR-1 (Pakistan Research Reactor-I) was used as gamma radiation source.

### Results

The Y doped ZnO nanostructures were characterized structurally using XRD, SEM, and EDS, the thermoluminescence performance was evaluated thermoluminescence dosimetry (TLD). Figure 2(a) represents the XRD patterns for different doped samples, the crystallite data for these samples is represented in Figure 2(b). The SEM images for the as-synthesized samples were performed for particle size and morphology analysis, as shown in Figure 3. The EDS was done to confirm Y doping (Figure 4). Finally, the photoluminescence and dosimetry characterization of the samples was done to evaluate the TLD performance of the samples. Figure 4(b) represents the Tyndall effect of the Y doped samples, Figure 5 shows the UV-Vis absorption and bandgap data whereas Figure 6 and Figure 7 demonstrate the TLD response of the samples after gamma irradiation.



## Discussion

XRD patterns of UCZ, CY0Z, CY5Z, CY7Z, and CY10Z NPs have been shown in figure 2(a). XRD patterns of these synthesized specimens are in agreement with the standard JCPDS (card no. 00-036-1451). There is no extra peak that depicts the presence of any external impurity or phase. The average crystallite size is plotted in Figure 2 (b). It is observed from the index pattern that peaks of doped materials (CY0Z, CY5Z, CY7Z, and CY10Z) got broadened as compared to uncoated ZnO peaks depicting reduction in crystal sizes which is in agreement with calculated crystallite sizes. It also demonstrates PEG's potential as a good surfactant for synthesizing nanocrystals of small size. The doped ZnO peaks have clearly shifted towards the lower  $2\theta$  value; the maximum shift was observed for CY7Z NPs. This is evidence of increased interplanar spacing thus marking successful doping of Y in ZnO lattice as  $Y^{+3}$  substitute and occupy  $Zn^{+2}$  ions site in lattice (Cheng et al., 2008).

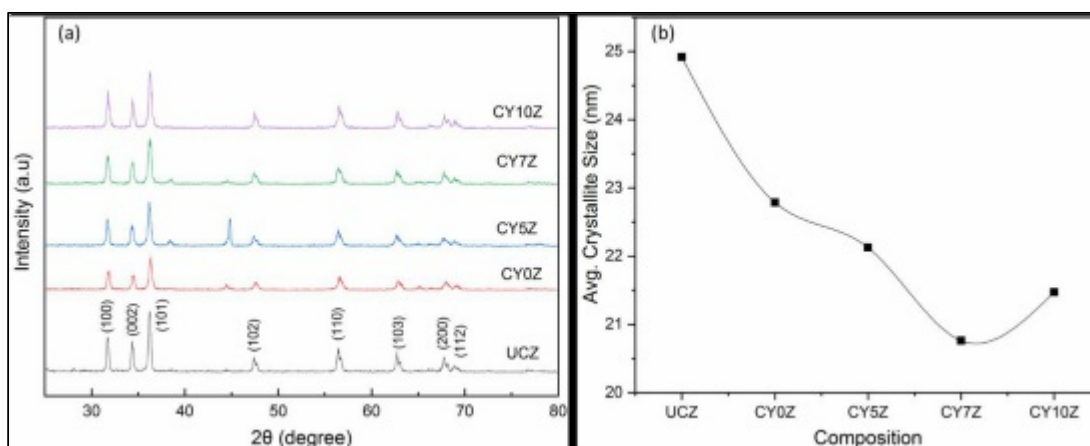


Figure 2: (a) XRD patterns and (b) average crystallite size of uncoated (UCZ), PEG coated Y doped ZnO NPs prepared hydrothermally (CY0Z, CY5Z, CY7Z, and CY10Z )

SEM micrographs of PEG-coated ZnO CY0Z, CY5Z, CY7Z, and CY10Z are shown in figure 3. Size of particle varied from 100-500nm, with morphology varying from irregular plates and rods to well-rounded plates and then needles. PEG-coated doped ZnO NPs with low doping concentration have small particle size of ~100nm while higher doping concentration leads to the larger size of ~400nm size and a plate and rod-like morphology. An increase in particle size can be attributed to the high concentrations of Y ions. This doping dependent change is morphology and size of NPs

has been observed before by many researchers (Zamiri et al., 2014)(Ishaq et al., 2013)(Shanmugam et al., 2015).

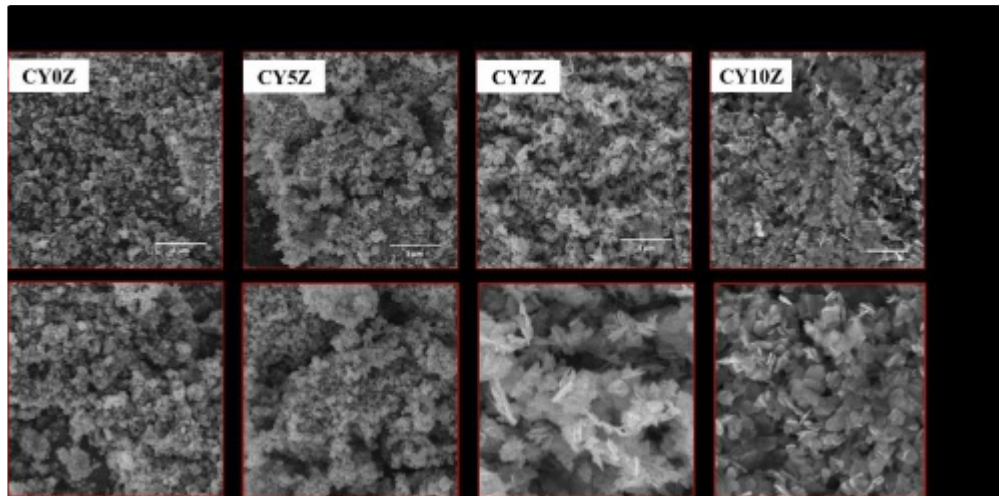


Figure 3: SEM images of CY0Z, CY5Z, CY7Z, and CY10Z NPs

Figure 4(a) shows an EDS study for 10 at% Yttrium doped ZnO nanoparticles material. EDS analysis confirmed the presence of only Zinc (Zn), Yttrium (Y), Oxygen (O) in the specimen. Tyndall test was performed for two doped samples (CY10Z and CY5Z). Tyndall effect shows very good colloidal stability of these NPs which attributes to the good PEG-Coating on these NPs.

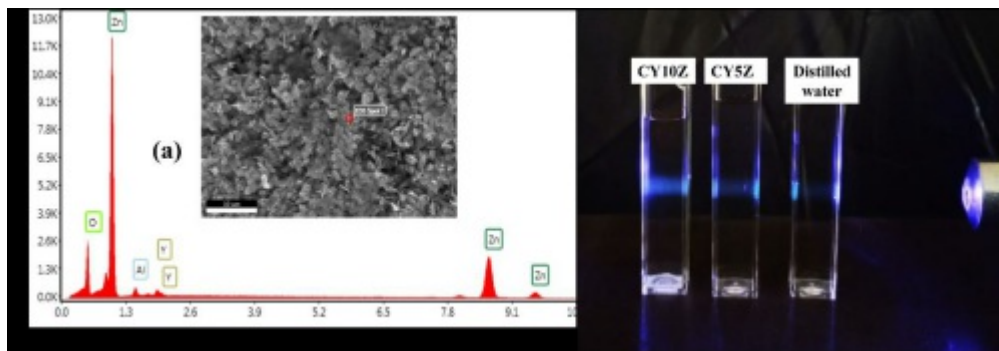


Figure 4: (a) EDS of 10 at% Y doped ZnO sample, (b) Tyndall effect for CY10Z and CY5Z NPs. UV visible spectra of CY0Z, CY5Z, CY7Z, and CY10Z NPs is shown in Figure 4(a). Characteristic absorption edge is observed at  $\sim 383$  nm, which is also a confirmation for the ZnO.<sup>[36]</sup> UV visible spectra exhibit a blue shift with an increase in the Y concentration as a dopant; indicating that the optical band gap has increased. Yttrium oxide ( $Y_2O_3$ ) has a larger optical band gap (5.5eV) as compared to pure ZnO ( $\sim 3.24$  eV).

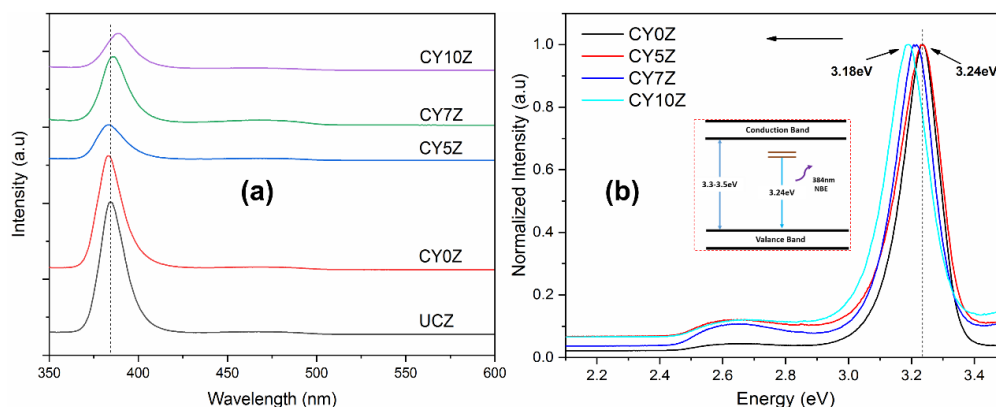


Figure 5. (a) UV-Vis spectra of prepared samples; (b) Bandgap energy plot from PEG-coated samples.

The sharp peak at  $434\text{cm}^{-1}$  shows the crystallization and wurtzite structure of ZnO crystals. This increase in optical band gap is then logically true as with doping of Y ions, optical band increases.<sup>[16]</sup> In actual, due to the presence of the Yttrium 4d levels in the conduction band (CB), some zinc (Zn) 4s and 4p levels will exchange with the forbidden band which consequently introduces impurity states (Wang et al., 2015).

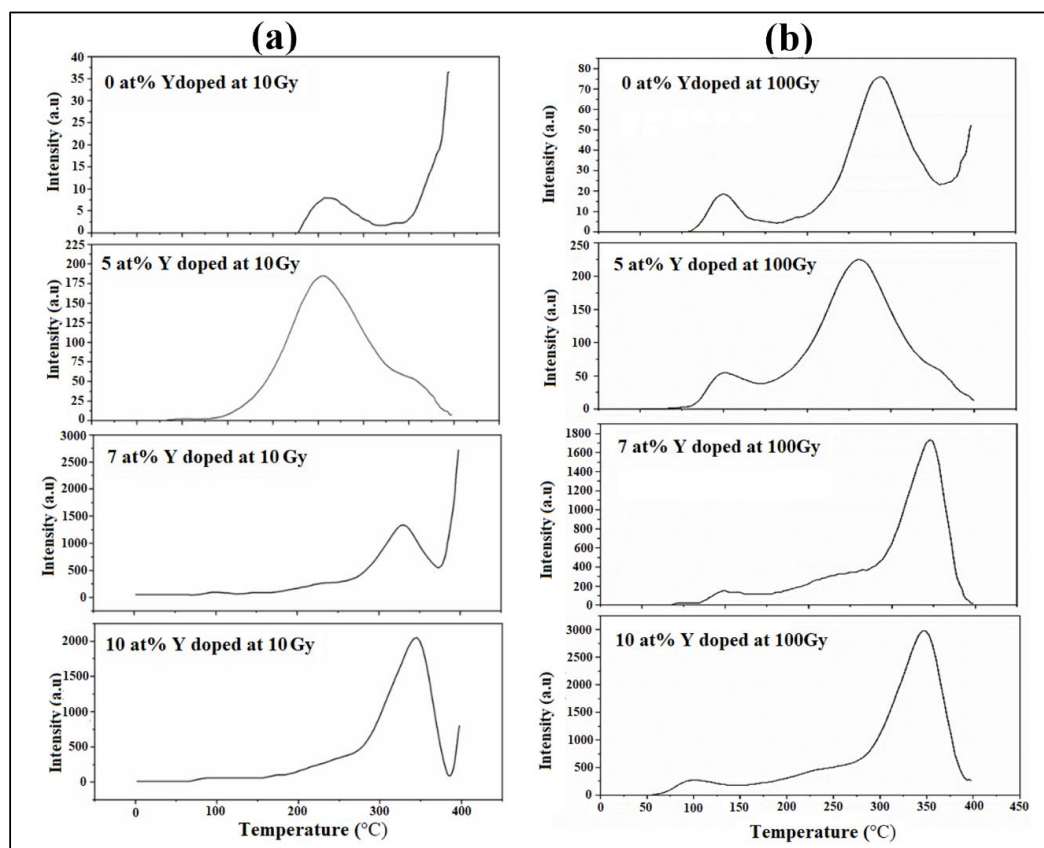


Figure 6: TLD response of Y doped NPs at (a) 10Gy dose (left column) and (b) 100 Gy dose (right column).

Thermoluminescence glow peaks for undoped and doped specimens have been shown in Figure 6(a), (b) for 10Gy, and 100Gy. Two broad peaks; at  $\sim 100^\circ\text{C}$  and  $\sim 250^\circ\text{C}$  were observed in the glow curves of CY0Z and CY5Z specimens. For CY7Z and CY10Z specimens peak shifted towards higher temperatures; observed at  $\sim 325^\circ\text{C}$ . If linearity is the concern, then Y ion concentration below 5 at% could play a role and can be optimized. TL phenomenon in pure ZnO is due to the presence of oxygen defects ( $\text{O}_i$ ). Reduction in particle size increases the number of surface states and consequently increases the amount of recombining charge carriers.

The enhancement in the thermoluminescence (TL) intensity is attributed to possible recombination centers generated as defects between valence and conduction band in  $\text{Zn}_{(1-x)}\text{Y}_x\text{O}$ . The forbidden bandgap is reduced for  $\text{Zn}_{(1-x)}\text{Y}_x\text{O}$  owing to the doping of Y ions. The 4d of yttrium states are set in the conduction band (CB), whereas the Zinc (Zn) levels 4s and 4p will move to the bandgap to generate defects. Furthermore, the ionic radius of  $\text{Y}^{+3}$  (0.9 Å) is slightly greater than that of  $\text{Zn}^{+2}$  (0.74 Å). When  $\text{Y}^{+3}$  replaces and occupies  $\text{Zn}^{+2}$  ions in the crystal lattice of ZnO, the space between neighboring planes expands, causing lattice distortion and multiple interstitial defects, which results in intermediate traps. This is also confirmed by the observed blue shift in UV visible spectrum. Thus a drastic increase in the radiative transitions occurs due to e-h pairs recombination (Wang et al., 2015, Thabit et al., 2021). At higher Y ions concentrations, TL response decrease with increasing gamma radiation dose. It can be attributed to fact that increasing dopant concentration, might lead to the pile-up of Y atoms and the creation of the Y-Y-O cluster at grain boundaries. This Y-Y-O boundary produces a quenching effect and kills electrons, consequently, reducing TL (Wang et al., 2015).

TL peak with maximum intensity is located at  $\sim 250^\circ\text{C}$  is the stable glow peak. Stable glow peak shifted towards higher temperature with increasing Y doping concentrations. The stability of this peak is mainly attributed to the presence of deep traps. Whereas the peak located at a lower temperature  $\sim 100^\circ\text{C}$  is owing to the shallow traps that lead to an unstable peak. This peak is of no importance for TL dosimetry applications as it will fade quickly and result in information loss.

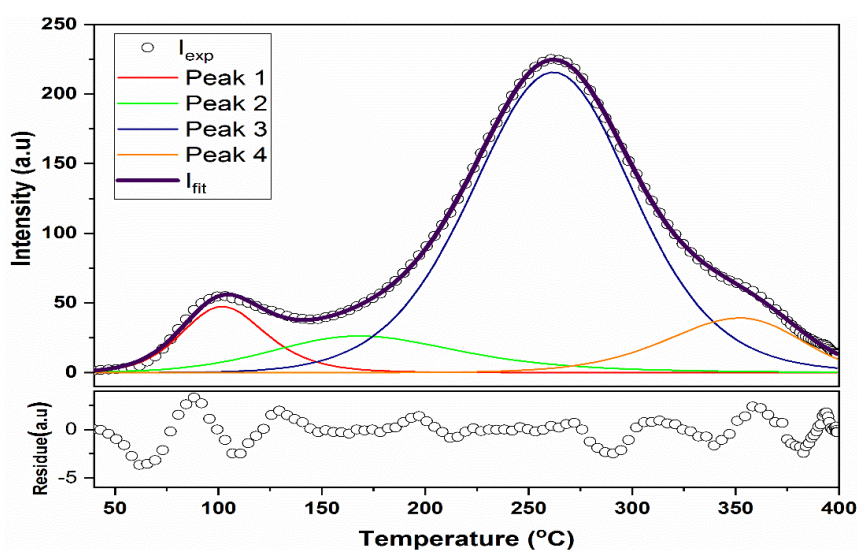


Figure 7: Deconvoluted peak of TLD response for 5 at% Y doped ZnO for 100Gy dose

Figure 7 shows the deconvolution of the representative glow curve of CY5Z at 100Gy to study the kinetics parameters of TL. It is displaying deconvoluted results obtained by applying the first-order kinetics (FOK) model. FOK results have a high figure of merit (FOM) = ~16% which is unacceptable (FOM must be ~1%). While the second-order kinetics (SOK) model gave 1.3% FOM. It shows  $Z_{(1-x)}Y_xO$  follows the second-order of kinetics (SOK) and there are a total of five small peaks within the broad peaks of the glow curve. These sub-peaks are providing information about the total number of traps present. Calculated kinetic parameters have been shown in Table 2. Though Yttrium doping improves the thermoluminescence intensity of ZnO however at higher Y concentrations (5 at%), it imparts a negative effect and deteriorates intensity at high doses.

Table 2: Determined kinetics parameters using computer program TL Analyzer

Parameter	Peak #1	Peak #2	Peak #3	Peak #4	Peak #5
TL Kinetic Model	SOK	SOK	SOK	SOK	SOK
$I_{max}$ (a.u.)	33.9	33.5	28.6	220.6	27.1
$T_{max}$ (°C)	93.3	116.5	175.9	260.4	355.2
$E_g$ (eV)	1.25	0.87	0.75	0.82	1.99
Frequency Factor ( $s^{-1}$ )	$1.75 \times 10^{16}$	$1.3 \times 10^{10}$	$1.1 \times 10^7$	$1.8 \times 10^6$	$5.1 \times 10^{14}$

## Conclusions

The uncoated pure ZnO and PEG (Polyethylene glycol) coated Yttrium (Y) doped ZnO was synthesized successfully via hydrothermal reaction. PEG coating proved a good surfactant and efficiently reduced particle size, but with increasing Y ion concentration beyond 5 at% particle size increased. PEG-coated Y doped nanoparticles exhibit variation in morphology from rod-like and plate-like and a mixture of these with size ranges in 100nm to 400nm. Y ion doping has altered the optical band gap of ZnO; the optical band gap increased with increasing dopant concentrations. Thermoluminescence response enhanced significantly with increasing Y doping. TL spectra displayed two broad glow peaks: at ~100°C (unstable) and ~250°C (stable peak). An increase in Y ion concentration enhances the TL intensity of stable peak and shift it towards higher temperature (~325°C). Doping concentrations above 5 at% led to compromise on linearity. Deconvolution of broad peaks indicated the presence of five different traps with trap depth 1.25, 0.87, 0.75, 0.82, 1.99 eV, in the Y doped ZnO which followed the second-order of kinetics. <sup>[0]</sup> In conclusion, Y doped ZnO nanoparticles were investigated which leads to the conclusion that these nanostructured materials are suitable for dosimetry applications.

## Declaration of Competing Interest

Nil

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