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SYNTHESIS AND PHOTOLUMINESCENCE PROPERTIES OF Tb3+doped Sr2P2O7 PHOSPHORS

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ABSTRACT

In this paper, synthesis and photoluminescence (PL) properties of rare earth Tb^{3+}doped$Sr_2P_2O_7$ Phosphors were prepared by conventional solid state reaction method and photoluminescence (PL) properties of samples were examined for proper excitation. All the samples were synthesized by high-temperature SSR- method. The X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy characterization confirms the formation of pure α-phase with crystallized in orthorhombic structure of samples. Surface morphology of the present phosphor has been studied by scanning electron microscope (SEM).The PL emission spectra of all samples exhibit characteristic green emission peaks of Tb^{3+} where the peak at 545 nm has the highest emission intensity for Tb^{3+} concentration of 5.0 mol%.The entire study reveals that the present phosphor has promising applications in the lamp industry especially for solid state lighting (mercury free excited lamp phosphor) and UV LEDs.

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INTRODUCTION

In the recent era of solid-state lighting the demand for mercury free excited phosphor-converted LEDs has significantly increased. Literature study authenticates that phosphate base phosphors are renowned for their thermal and chemical stability. Since, they are extensively used as hosts doped with different rare earth ions to obtain phosphors for verity of lighting applications[1]. Consequently the research on phosphors suitable for fabricating NUV excited light-emitting diodes has attracted more attention. Compared to the conventional incandescent lamps, LEDs have numerous advantages such as less energy consumption, optimum luminosity, and extended lifetime. They have been used mainly for LCD backlighting, traffic lights, and information boards. In sight of green emission at around 545 nm trivalent terbium ions based phosphors play a vital role in the field of displays and enlightenment [2]. Particular phosphors such as $GdMgB_5O_{10}$, Y_2SiO_5 and $LaPO_4$ based on energy transfer from Ce^{3+} to Tb^{3+} found diversified applications for their green emission in lamps. Herein, the main objective of this work is to develop an efficient pyrophosphate phosphor that absorbs energy in the near-UV range and emit in the visible range and to examine the photoluminescence and structural properties of the newly synthesized $Sr_2P_2O_7$ pyrophosphate phosphor. The prepared phosphor has been characterized by XRD and SEM. The photoluminescence investigations are performed to explore the luminescence characteristics of trivalent terbium doped strontium pyrophosphate [3-5].

The observed blue and intense green emission from $Sr_2P_2O_7$:Tb^{3+} with 232nm (mercury free) excitation suggests a possibility of applying the newly synthesized phosphors for phosphor- converted UV-LEDs and solid state lighting. The luminescence from the phosphors can acquire by exciting the phosphor in different excitation radiations such as UV and visible radiation (photoluminescence), a beam of energetic electrons (cathodoluminescence), x-rays (x-ray excited luminescence), etc. Luminescence measures the energy levels of the luminescence centers formed inside the phosphors. The energy level of a luminescence center is defined as its characteristic state, which is related to the physical nature of the center and to the energetic and dynamic processes that the center undergoes. Rare earth doped inorganic luminescent materials are well known for emission in distinct wavelengths in the electromagnetic spectrum [6]. The phosphors have a wide range of applications in the lighting devices such as cathode ray tubes (CRT), tri-phosphor fluorescent lamps, x-ray intensifying screens and newly developed vacuum mercury-free lamps. It has other applications in a display such as plasma display panels and field emission displays. Finally, it is concluded that the different inorganic pyrophosphates were showsvery good luminescent behavior [7-10]. They were adopted in different type of preparative methods were used to prepare the pyrophosphate phosphors, but the majority were synthesized by conventional solid-state method, it is very easy, bulk materials were prepared and need not required sophisticated equipment and also non-toxic in nature. Therefore, in this paper reported, synthesis and photoluminescence properties of

Tb^{3+}doped $Sr_2P_2O_7$ pyrophosphate phosphors were successfully synthesized by conventional SSR-method fired at 1200°C for 3hours. The prepared phosphors were characterized by subjected to PL, XRD, SEM and FTIR analysis factors were affecting on the structure and performance of the phosphors were studied.

EXPERIMENTAL METHOD

The conventional Solid state reaction method was utilized for preparing these phosphors, which is the simpler and standard method. The inorganic compounds like Strontium carbonate ($SrCO_3$), Ammonium phosphate $(NH_4)_2HPO_4$and Terbium Oxide (Tb_2O_3) (A.R) of high purity (Sigma Aldrich 99.9%) chemicals were used as starting materials. First, we prepared $Sr_2P_2O_7$pyrophosphate phosphor, without adding any dopants, as a host material, by weighing Strontium carbonate ($SrCO_3$), Ammonium phosphate $(NH_4)_2HPO_4$in stoichiometric proportions of 2:1.

The basic chemical reaction for Sr2P2O7 pyrophosphor can be described as follows:

$Sr(CO3) + 2((NH_4)_2HPO_4) \rightarrow Sr_2P_2O_7 + 2NH_3 + 3H_2O + 3CO_2$.

The compounds were mixed with a spatula and then ground into a fine powder using an agate mortar and pestle for 30 minute to obtain homogeneous mixture.The homogeneous mixture was transferred into alumina crucible and put inside the muffle furnace for heat treatment at 1200°C in the air for 3 hours in a muffle furnace with a heating/cooling rate of 5°C/min. After heating treatment, the furnace was allowed to cool naturally to the room temperature. All prepared samples were in the pure white powder form. The samples were grinded in a mortar–pestle for half hour to obtain the homogeneous fine powder form. Similar procedure is been followed for Tb^{3+} doped $Sr_2P_2O_7$pyrophosphate phosphors. All phosphors were successfully synthesized in pure powder form [11-13].

To identify the crystal phase, XRD analysis was carried out with a powder diffractometer (Rigaku-D/max 2500) using Cu Kα radiation. The Photoluminescence (PL) emission and excitation spectra were measured by Spectro fluorophotometer (SHIMADZU, RF-5301 PC) using a Xenon lamp as an excitation source. All the spectra were recorded at room temperature. The morphologies (SEM) of the phosphor powders were obtained by using the Nova NanoSEM450.FTIR analysis analytical techniques were used by (JASCO–4600) for the starching and bending analysis of the samples. Fourier transform infrared (FTIR) spectra of the samples were recorded by FTIR-4100 type A IR spectrometer in transmittance mode in wave number range of 400–4000 cm^{-1}.

RESULTS AND DISCUSSION

Crystal structure analysis: The XRD patterns of Tb^{3+}doped $Sr_2P_2O_7$ samples are compared with JCPDS card no. 24-1011have nonoticeable influence on the crystalline structure of the host $Sr_2P_2O_7$phosphors as shown in figure.1. The hkl parameters of the prepared samples are similar to that of the JCPDS card no. 24-1011 of α-$Sr_2P_2O_7$ that indicates the existence of a pure single-phase α-$Sr_2P_2O_7$phosphor. The structural parameters of the samples were analyzed using powder software for XRD analysis. The analysis confirms that the samples have a pure α-phase with crystallization in the orthorhombic structure and space group of P_{nam}. The doping of various Tb^{3+} ions and with their different (mol%) concentrations does not make any perceptible variation in XRD patterns as well as no other peaks found than that of $Sr_2P_2O_7$ in these patterns. This indicates that the prepared samples are single phased and substitution of Sr^{2+} by Tb^{3+} does not cause any substantial modification in the crystal structure of host [14]. Some prominent intense peaks are observed in the XRD patterns of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations at different 2θ values of 12.12°, 25.95°, 26.27°, 26.93°, 33.16°, 33.63° and 44.40° corresponding to the (110), (201), (031), (211), (002), (141) and (232) planes for these

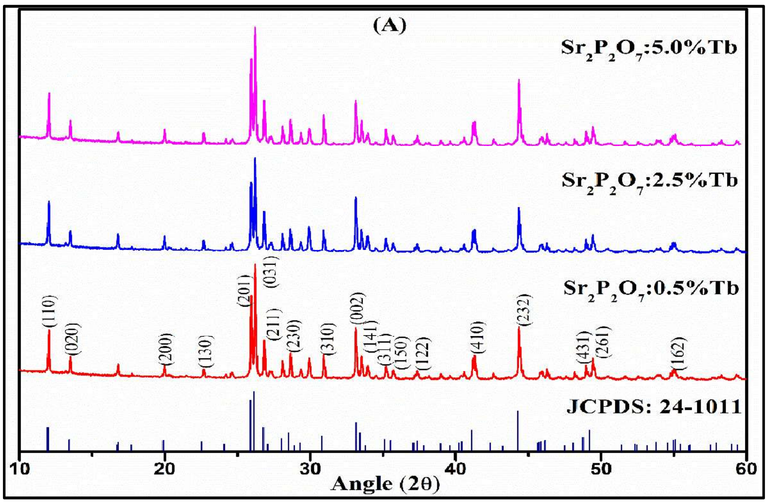


Figure 1. XRD patterns of Tb^{3+}doped $Sr_2P_2O_7$phosphor with different (mol%) concentrations and JCPDs Card No. 24-1011

Table-1. Crystal structure parameters of pure $Sr_2P_2O_7$phosphor and JCPDS (24-1011) Data

JCPDS (24-1011) Data			Experimental Data of pure $Sr_2P_2O_7$ phosphor		
a = 8.917 Å	b = 13.160 Å	c = 5.400 Å	a = 8.972 Å	b = 13.254 Å	c = 5.546 Å
V = 633.677 $Å^3$			V = 654.659 $Å^3$		

Table-2. Crystal structure parameters &Crystallite Size ofTb^{3+}doped $Sr_2P_2O_7$phosphor with different(mol%) concentrations

ample	Lattice Parameter			Volume of Unit Cell	Crystallite Size (nm)
	a (Å)	b (Å)	c(Å)	‘V’ ($Å^3$)	
$Sr_2P_2O_7$: 0.5 mol% Eu^{3+}	8.864	13.186	5.531	645.13	71.87
$Sr_2P_2O_7$: 2.5 mol% Eu^{3+}	8.855	13.157	5.432	634.11	66.13
$Sr_2P_2O_7$: 5.0 mol% Eu^{3+}	8.846	13.135	5.365	627.19	63.73

host lattice respectively. Sharp intense peaks in the XRD patterns of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations signify that all samples were polycrystalline structure [15]. Detailed lattice parameters of pure $Sr_2P_2O_7$phosphors with calculated values from the experimental values using powderX software and the values reported in literature are summarized in Table.1. The calculated Crystalline parameters and Crystallite size of Tb^{3+}doped $Sr_2P_2O_7$ phosphors with different (mol%) concentrations were shows in Table.2.The average crystallite size of Tb^{3+} doped $Sr_2P_2O_7$phosphors with different (mol%) concentrations were calculated by using the Scherrer's formula is$D=K\lambda/\beta\cdot cos\theta$. Where D is the average crystallite size or ordered domain (in nm), λ is x-ray wavelength (λ= 1.54178 Å), βis the full-width half maximum (FWHM) of the intense peaks of host lattice, and K is the shape factor (K = 0.9). From the table it observed that the lattice parameter values, unit cell volume and crystallite size decrease as on the Tb^{3+} doped $Sr_2P_2O_7$ phosphors with different (mol%) concentrations increases, which is suggest that the incorporation of doing ion increases as at higher temperature.

Photoluminescence analysis: Figure 2. shows the excitation spectrum of Tb^{3+} doped $Sr_2P_2O_7$phosphors were recorded bymonitoring the phosphor by green emission at 547 nm. The excitation spectra display only single band centered at 232 nm which could be ascribed to the $^7F_6 \rightarrow {}^9E$ electronic transitions, that can be attributed to the $4f^8 \rightarrow 4f^7 - 5d^1$ spin allowed the transition of Tb^{3+}. Whereas the ^9E states can come from the splitting of $4f^7$ 5d state in the crystal field with D_2 symmetry inside the host. An excitation band illustrates that the phosphor can be excited through the UV light of about 232 nm. Generally, the intensity f–d transitions is much intense compared to that of the f–f transitions for low concentration of doping of Tb^{3+} ion. Although the intensity of f–d transitions is much weaker compared to that of the f–f transitions for a high concentration of doping of Tb^{3+} concentration.

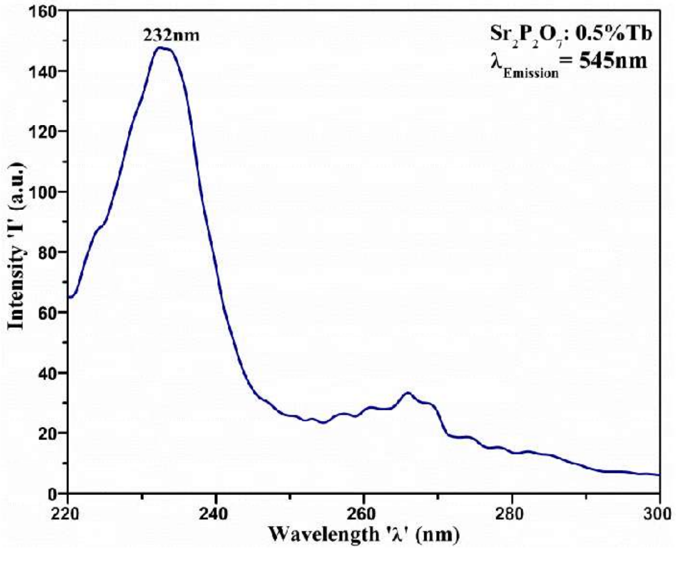


Figure 2. PL excitation spectra of Tb^{3+}doped $Sr_2P_2O_7$phosphor for $\lambda_{Emission}$= 545 nm

This could come about because of the f–f transitions are forbidden transition while the f–d transitions are allowed according to the Laporte's rule [100]. PL emission spectra of all Tb^{3+} doped $Sr_2P_2O_7$phosphors samples were monitored by exciting peak maximum wavelength i.e., 232 nm to determine the optimum PL emission of the doped phosphors as well as the preparation process based for different concentration of doping ion [16-18]. Figure 3.shows PL emission spectra of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations for $\lambda_{Excitation}$= 232 nm. PL emission spectra demonstrate the several sharp peaks centered at 415, 436, 469, 491, 545 and 584 nm as a consequence of the characteristic's radiative transitions of Tb^{3+} ion which can be attributed to the electron transitions taking place correspond to the $^5D_3 \rightarrow {}^7F_5$, $^5D_3 \rightarrow {}^7F_4$, $^5D_3 \rightarrow {}^7F_3$, $^5D_4 \rightarrow {}^7F_6$, $^5D_4 \rightarrow {}^7F_5$, and $^5D_4 \rightarrow {}^7F_4$, respectively. PL emission spectra consist of two groups of emission lines; first group of lines is located in the wavelength range 400 – 475 nm which is consigned to the $^5D_3 \rightarrow {}^7F_J$ (J = 5, 4, 3) electron transitions of Tb^{3+}, second group of lines are located in the wavelength range of 475 – 600 nm which is resulting due to $^5D_4 \rightarrow {}^7F_J$ (J = 6, 5, 4) electron transitions of Tb^{3+}. It is found that the emission takes place due to $^5D_4 \rightarrow {}^7F_J$ (J = 6, 5, 4) transitions are much stronger than that of $^5D_3 \rightarrow {}^7F_J$ (J = 5, 4, 3.) transitions

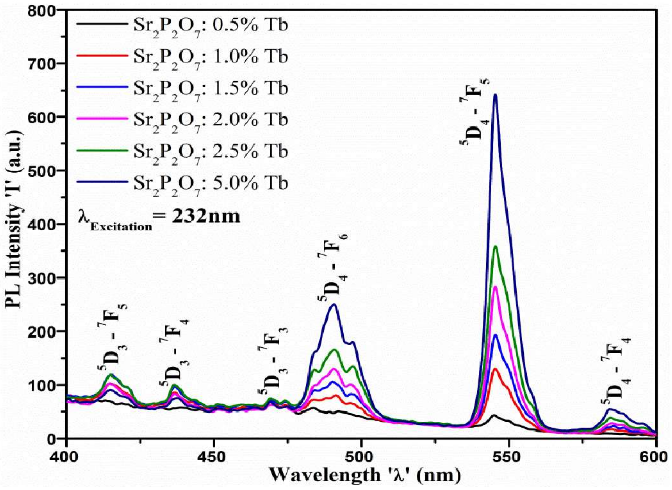


Figure 3. PL emission spectra of Tb^{3+}doped $Sr_2P_2O_7$phosphor with different (mol%) concentrations for $\lambda_{Excitation}$= 232 nm.

Sharp emission attributed to the predominant $^5D_4 \rightarrow {}^7F_5$ transition at about 545 nm which can be resulting due to the most probable electron transition take place in Tb^{3+} ion. PL emission spectra Tb^{3+} doped $Sr_2P_2O_7$ phosphor illustrate that as the doping concentration Tb^{3+} is increased the PL emission intensity in the range 400 – 475 nm increases poorlyas compared to that of the PL emission intensity in the range 475 – 600 nm. The reduction in PL intensity observed in level $^5D_3$ as the concentrations of Tb^{3+} ion increases can give the evidence for the presence of energy transfer due to cross-relaxation take place between level $^5D_3$ and $^5D_4$. In the phenomenon of the cross-relaxation energy transfer process, the excited ion transfers its partial energy to another ion. This process generally observed in Tb^{3+} ion doped phosphors where the energy transfer occurs between two Tb^{3+}ions situated at a certain distance, as the Tb^{3+} – Tb^{3+} ion distance increases the cross-relaxation mechanism reduces [19, 20]. The distance between to ions mainly depends on the host lattice structure. As explained in x-ray diffraction of rare earth doped $Sr_2P_2O_7$ phosphor, the reduction is observed in lattice constants and the crystallite size of the phosphor due to the increase in the concentration of doping ion. That implies as the doping concentration increases the accumulation of doping ion in the phosphor increases and the crystallite size decreases which can result in the large density of doping ion [21-23]. Therefore, the possibility of cross-relaxation interaction between two close ions immensely increases. In cross-relaxation process, the energy difference between the two excited states $^5D_3$ and $^5D_4$ is approximately equal that of the energy difference between the $^7F_6$ ground state and higher ^7F_J states. The occurrence of cross-relaxation would intensify the population of the 5D4 state and as a result, the minimization of PL intensity of $^5D_3$ compare to PL intensity of $^5D_4$.

FTIR analysis: FTIR transmittance spectra of Tb^{3+}doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations were synthesized by conventional solid state reaction method were depicted figure.4. The FTIR spectra of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations were recorded for the wavenumber range from 400 – 4000 cm-1, the fingerprint region of the FTIR spectra of the range 400–2000 cm-1 has been demonstrated in the graph. All FTIR spectra were recorded from the KBr pellet of $Sr_2P_2O_7$. The KBr pellet for all samples were prepared for 99:1% ratio of KBr to $Sr_2P_2O_7$. The thickness of each pellet is approximately maintained up to 1 mm which has semi-transparent nature. Various transmittance bands of pyrophosphate group and phosphate group are observed in the FTIR spectra. FTIR spectra of the phosphors show the evidence of the multiple vibrational bands. The pyrophosphate group $(P_2O_7)^{4-}$ could be the form of O_3P-O-PO_3, and can be attributed to an assembly of the vibration modes of the PO_3 groups and the P-O-P groups [24-25]. The formation of the $P_2O_7^{4-}$ modes in the phosphor is conceded on the basis of the PO_3 and P-O-P vibrations. From the figure.4 it is observed that the FTIR spectra of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations were consistent for all samples and there is no change in peak position. The FTIR spectra of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations shows characteristic bands at 433.90, 486.94, 500.43, 563.11, 620, 746.31, 974.84, 1014.37, 1047.16, 1062.58 and 1190.83 cm-1.

SEM analysis: The SEM images of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations were synthesized at 1200^0C annealing temperatures only few concentrations are shows in Figure 5. The powder form of Tb^{3+} doped $Sr_2P_2O_7$ phosphor materializes in highly crystalline form with uneven morphology observed under different resolution and the average grain size of 2-10μm as seen in SEM images. The images revealed that the phosphor have very porous structure of particles and it has possessed foamy nature or cotton-like structure due to the highly agglomerated particles. It is observed that the crystallites particles having non-uniform shape with evolved boundary of submicron size and strongly agglomerated [27-30].

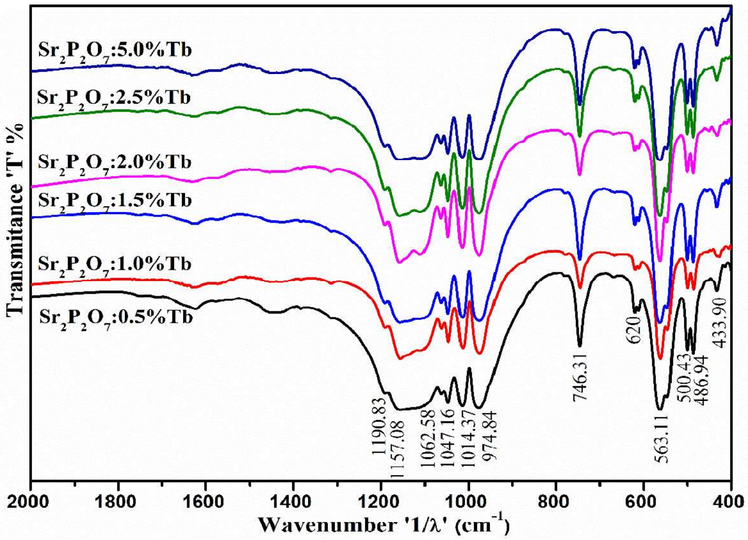
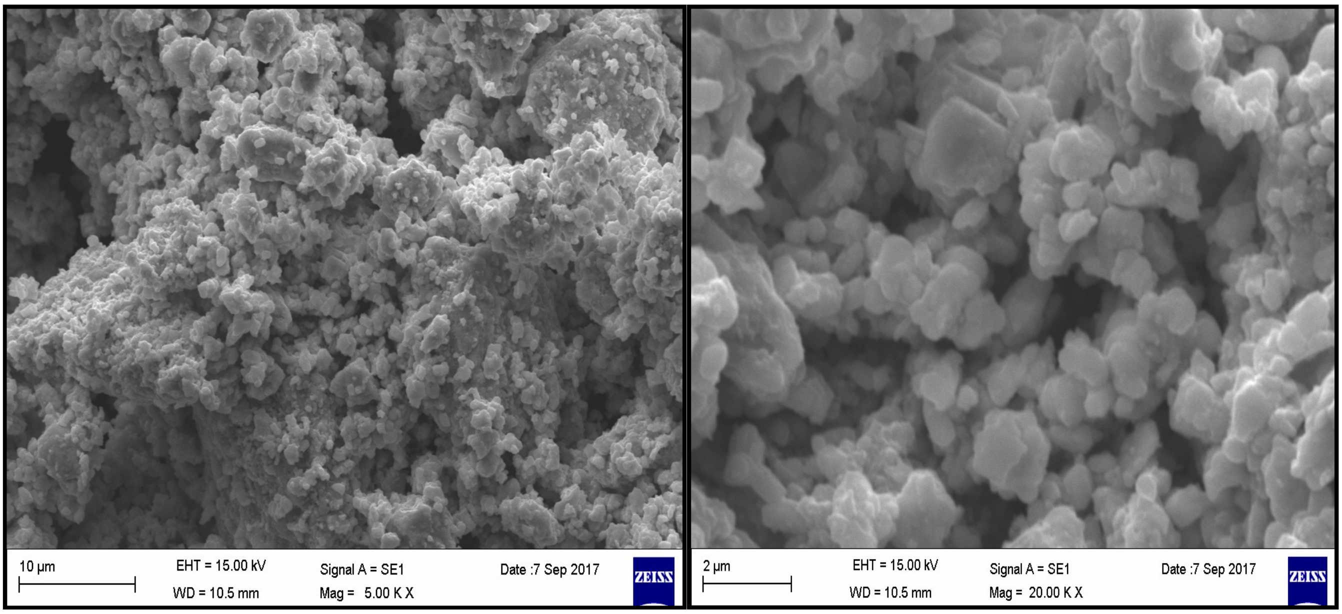


Figure 4. FTIR spectra of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations



(a) (b)

Figure 5. SEM images of Tb3+ doped Sr2P2O7 phosphor with different (mol%) concentrations

The intense peak attributed to the P-O-P symmetric stretching vibration mode is been observed at 746.31 and 974.84 cm-1 in the IR spectrum. It could be assigned to the symmetric stretching of the P-O-P bonds in the $(P_2O_7)^{4-}$ group. The FTIR bands are manifest characteristic of the vibration modes of the pyrophosphate and phosphate groups in the phosphors [26]. Thus, from the FTIR results it is found that the formation of phosphors was consistent and having uniform crystal phase formation as analyzed in x-ray diffraction.

CONCLUSIONS

The photoluminescence studies for Tb^{3+} doped $Sr_2P_2O_7$ phosphors displayed fascinating results, the following are the logical conclusions:

- In conclusion, we successfully synthesized by conventional solid state reaction method has been followed by Tb^{3+} doped

$Sr_2P_2O_7$ phosphor with different (mol%) concentrations were synthesized at 1200^0C annealing temperatures for 3 hours. In this process, the reaction temperature for the formation of $Sr_2P_2O_7$ phosphor is found to be lower compared to other methods and this is one of the important results of the present work.

- The method followed has several benefits such as low cost, energy efficiency, high production volume, no toxic in nature, do not require sophisticated equipment's and above all, easy method of preparation. The PL of the Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations has been observed and analyzed.
- The excitation spectrum of Tb^{3+} doped $Sr_2P_2O_7$phosphors were recorded by monitoring the phosphor by green emission at 547 nm. The excitation spectra display only single band centered at 232 nm which could be ascribed to the $^7F_6 \rightarrow {}^9E$ electronic transitions, that can be attributed to the $4f^8 \rightarrow 4f^7$ - $5d^1$ spin allowed the transition of Tb^{3+}ions.
- PL emission spectra of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations for $\lambda_{Excitation}$= 232 nm. PL emission spectra demonstrate the several sharp peaks centered at 415, 436, 469, 491, 545 and 584 nm as a consequence of the characteristic's radiative transitions of Tb^{3+} ion which can be attributed to the electron transitions taking place correspond to the $^5D_3 \rightarrow {}^7F_5$, $^5D_3 \rightarrow {}^7F_4$, $^5D_3 \rightarrow {}^7F_3$, $^5D_4 \rightarrow {}^7F_6$, $^5D_4 \rightarrow {}^7F_5$, and $^5D_4 \rightarrow {}^7F_4$, respectively.
- The energy transfer comes about to the cross-relaxation resulting in strong green emission, which can reveal the doping ion Tb^{3+} substitutes the host Sr^{2+} ion sitewithout any structural deformation.
- The FTIR spectra of Tb^{3+} doped $Sr_2P_2O_7$ phosphor with different (mol%) concentrations shows characteristic bands at 433.90, 486.94, 500.43, 563.11, 620, 746.31, 974.84, 1014.37, 1047.16, 1062.58 and 1190.83 cm-1. The intense peak attributed to the P-O-P symmetric stretching vibration mode is been observed at 746.31 and 974.84 cm-1 in the IR spectrum.
- The powder form of Tb^{3+} doped $Sr_2P_2O_7$ phosphor materializes in highly crystalline form with uneven morphology observed under different resolution and the average grain size of 2-10µm as seen in SEM images.

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