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Optical Properties of Sm^{3+} and Nd^{3+} Activated Strontium Calcium Pyrophosphate Phosphor Solid State Lighting

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Abstract

A series of Sm^{3+} and Nd^{3+} activated $SrCaP_2O_7$ pyrophosphate phosphors were combined by solid state diffusion technique. The X-ray diffraction (XRD) and photoluminescence (PL) properties of these phosphors were studied at room temperature. The excitation spectra indicate that these phosphors can be effectively excited by mercury-free excitation. Excitation spectrum of $SrCaP_2O_7$:Sm^{3+} phosphors viewed at 564 nm and excitation observed in the 300 nm to 450 nm wavelength region. And the emission spectrum sequences from 550 to 650 nm, with the main peaks located at 564 nm, 602 nm, and 647 nm, resemble transitions $^4G_{5/2}\rightarrow{}^6H_{5/2}$, $^4G_{5/2}\rightarrow{}^6H_{7/2}$. Whereas, the excitation spectrum of $SrCaP_2O_7$:Nd^{3+} reveals that the doped samples have a sharp UV excitation peak located at 437 nm, which is attributed to band-to-band transitions. It is quite interesting to note that in the emission spectra of $SrCaP_2O_7$:Nd3+ phosphors, the emission from the host material at 708 nm corresponds to the $I_{9/2}\rightarrow H_{11/2}$ absorption band of N^{d3+}. The Fourier transform infrared spectrum (FTIR) was scanned at room temperature and the surface morphology was examined by scanning electron microscopy (SEM). The prepared phosphor exhibited intense orange red emission and near infrared region with Hg-free excitation, which is advantageous in solid-state illumination excitation. Therefore, the availability of such a phosphor would benefit significantly in the development of solid-state lighting applications.

Keywords: Photoluminescence, Pyrophosphate, Phosphors, Solid State Lighting, SEM, FTIR, XRD

1. Introduction

Luminescence is “cold light”, light from other energy sources, which can occur at normal and low temperatures. In luminescence, a certain energy source kicks an atomic electron out of its earth state into a contented state, and then the electron returns energy in the form of light. So the word phosphor comes from the Greek language and signifies light carrier, meaning light. -drive or luminescence returns to its original state. Luminescent material is also called 'phosphors'. The term ‘cent materials; Barium sulphide is one of the naturally occurring phosphors. Phosphor is luminescent, which means it releases energy into a contented electron like light. Electron excitation is caused by the absorption of energy from an external source such as another electron, photon or electric field.

Lanthanides is a rare earth metal found in the sixth period of the periodic table, and contains elements of La, Ce, Pr, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu [1]. Lanthanide ion doped materials such as luminescence have been widely used in technical intellectual, lasers and scintillator. Earth borophosphates with rare alkali are among the most frequently studied subjects due to showing both structural variability and important luminescence properties. Alternatively, rare earthy objects with a deep spectral hole can be a complete candidate for optical data retention of the highest memory device [2]. The rare earth of the lanthanide group has been implicated in $CaBPO_5$ in previous reports [4 - 6]. A new approach to this study is to use rare earth metals such as Y, Er, Gd, La, Nd in CaBPO5 for solid state reactions, as well as to detect bright objects with sufficient radiation to form candidates as non-linear optics. In addition, the structures of the crystal structure were resolved by the Rietveld filtration method using X-ray powder diffraction patterns at that time to our knowledge.

Rare Earth doped phosphors is one of the luminescent constituents of white LEDs. It is well known that choosing the right host matrix is important for rare earth phosphors, because the crystal formation of the right host can have a significant impact on the efficiency of photoluminescence ions of earth activator ions are rare [7]. Many compounds of oxysalts have been used, such as phosphor-containing substances eg, vandate [8, 9], molybdate [10], and so on. Among the many inorganic substances, phosphates are widely used as a potential source of phosphors due to their high thermal conductivity and chemical stability. Research into the efficacy of photoluminescence of phosphate-based compounds in rare earth cations has contributed to the formation of other novel phosphors. Chen et al. [11] reported a new phosphor $NaAlP_2O_7$: Eu^{2+} with a bright green light that appears to be active, which can be illuminated with NUV LED chips. This is the first example of a phosphor family based on $NaAlP_2O_7$ that is doping with rare earth ions. To test new visible red light phosphors, Eu^{3+} ions, as a dopant agent with multiple 4f electronic variables, usually incorporated into various handling materials.

Phosphorescence is a phenomenon of luminescence, that is, it emits light when exposed to radiation such as UV or electron beam ,since the discovery of phosphors has attracted a lot of interest due to its various dynamics and commercial interactions in several fields such as LEDs, lasers , non-mercury-free fluorescent lamps, plasma display panels (PDPs), luminescent films, OLEDs, polymeric optical amplifiers, solar cells, MRI comparison agents, display devices in the solid state power plant [12]. In addition, many phosphors-containing substances are commonly used in our daily lives, at home, in the laboratory and in hospitals etc. Some of the equipment containing phosphors items are television screens, fluorescent bulbs, computers and cell phones etc. Phosphor compounds of solvents such as oxides, nitrates, phosphates, and silicates with rare earth or metallic metals such as ion activators, interact less with the lattice of the host due to the involvement of f-f modification and the involvement of d-electron. Good phosphors is a nano \ micro-sized substance that absorbs energy and evaporates as fast as electromagnetic radiation. The electromagnetic radiation emitted by phosphor is usually in the visible environment \ UV.

In recent years, the orthophosphate family has solid-state white light-emitting diodes (LEDs). The orthophosphate group, PO_4, is the most common phosphors-based anion. Ions, HPO_4 H_2PO_4 are also found in many phosphors compounds. Normally all four oxygen atoms are normally linked to cations in solid phosphors leading to tightly bonded crystal structures. Many negative phosphates (PO_4) are connected to a transition or internal transformation metal that contains excellent charge when with ionic letter M-O it later became a compound character for the rise of the P-O bond. Phosphates containing lanthanum ion have a good surface and high melting points compared to other traditional phosphate substances. In addition rare earth phosphates comprising lanthanide ores and phosphate groups are phosphate compounds widely used in various fields such as ion exchange, fluorescence, catalysis and gas sorption etc.

2. Experimental

2.1 Synthesis Techniques of Phosphor Materials

With regard to the chemical composition of phosphors, one might think that the synthesis of luminescent phosphor should be as simple as most handling materials are known and actually present. In contrast, the synthesis of phosphors with desirable physiological properties is a bit difficult. Difficulty arises as one has to consider a few factors such as the combination of traps on selected sites, removal of residual impurities, the exact amount of grain and the genetic makeup appropriate for the application, production costs, batch homogeneity and reproduction, etc. To date to make things easier to synthesize phosphors, there are many advanced methods, some of which are not well-targeted termed as 'Novel Synthesis' (e.g. fire compounding). Novel syntheses compensate for the non-stressful mechanisms of phosphor synthesis and often make it possible to control particle size, phase formation and structure. The different combinations of methods are listed in Table 1. All of these methods are not used in the current study. The method used in this work is therefore described in detail for example.

Sr. No.	Technique	Synthetic Approach
1	Wet - Chemical	i. Co-Precipitation
		ii. Re-Crystalisation
2	Solid state	i. Melting

		ii. Solid state diffusion method
3	Novel Synthesis	i. Molten salt
		ii. Solvothermal method
		iii. Sol-gel
		iv. Hydrolytic sol-gel
		v. Non-Hydrolytic sol-gel
		vi. Pechini and citrate gel method
		vii. Polymer pyrolysis
		viii. Spray pyrolysis and sonochemicals
		ix. Cryochemicals synthesis including freeze drying
		x. Solid state metathesis

Table 1: List of various methods for preparation the phosphors

2.2 Solid state diffusion Technique:

Solid performance distribution is the process by which liquid, gas, or other solids can combine with a solid conductor at the atomic level, in order for the dispersal to occur, there must be a concentration and concentration at the dispersion process that may be affected by an increase in temperature. Figure 1 represents a flow chart for a solid state distribution.

The chemicals used in these methods are given below,

Host material:

Strontium carbonate ($SrCO_3$) = 147.63
Calcium carbonate ($CaCO_3$) = 100.09
Ammonium phosphate ($NH_4H_2PO_4$) = 115.03

Dopant (rare earth ions)

Samarium (Sm) = 348.72
Neodymium (Nd) = 336.5
Ammonium cerium nitrate (((NH_4)2Ce $(NO_3)_6$) = 548.23

Ex. Solid state diffusion synthesis of SrCaP2O7: Nd pyrophosphate phosphor

Weight of strontium carbonate $SrCO_3$ = 0.9735 g
Weight of Calcium carbonate ($CaCO_3$) = 2.00 g
Weight of ammonium orthophosphate ($NH_4H_2PO_4$) = 1.53 g
Concentration of Dopant and its weight 0.1%, 0.3%, 0.5%, 1%
The chemical reactions is as follows:

$$Sr(CO_3) + Ca(CO_3) + 2(NH_4H_2PO_4) \rightarrow SrCaP_2O_7 + 2NH_2 + 3H_2O + 2CO_2$$

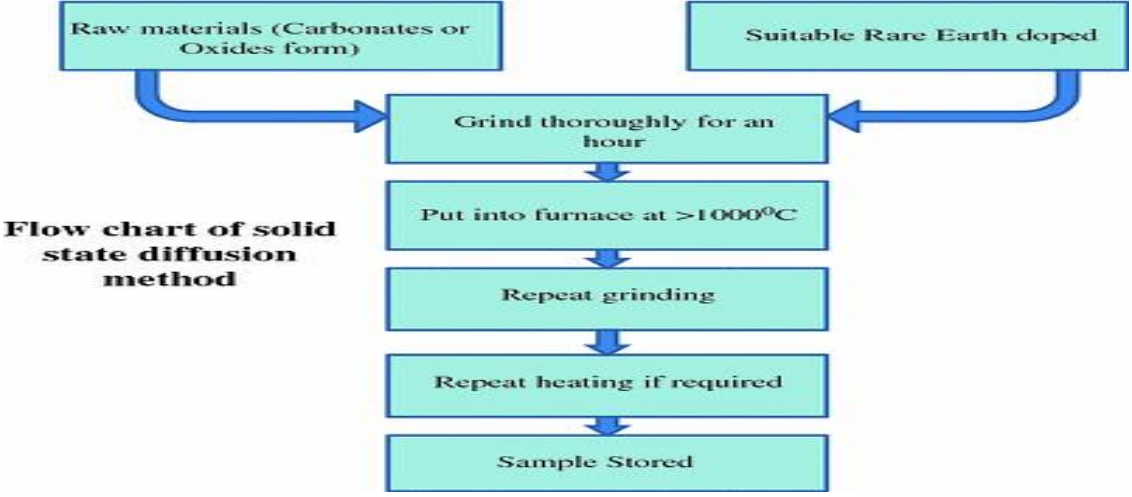

Figure 1 The flow chart of solid state diffusion.

3. Results and Discussion

3.1 X-ray diffraction pattern and structural behavior of $SrCaP_2O_7$phosphor

Research on pyrophosphor has become a hot concern in the discovery of new phosphor materials that have also proven proficient in the application of light conversion phosphors for white LEDs. Phase purity of the sample was checked by powder XRD using a PAN-analytical diffraction meter (Cu-Ka radiation) at a scanning step of 0.010; persistence time 20 s, 2θ range 100 to 800. Recently, Kim et al described Europium-activated $SrCaP_2O_7$ nanophosphors. The XRD configuration of $SrCaP_2O_7$ is shown in Figure 1. X-ray powder diffraction (XRD) measurements were performed to conclude the phase purity of the prepared materials. For the acquired stage, it is carefully perceived that there are twenty-seven raw material peaks. Unable to access standard JCPDs file of $SrCaP_2O_7$ material. However, we associated the current material with nanophosphors described in the US patent [13] and it gets better agreement and shows the fine linkage. The change in temperatures does not result in the new phase, except for the unidentified fundamental phase. Therefore, we anticipate that the acquired indefinite phase will likely be a new phase. Admiring this point, a preliminary examination remains. This result modestly reveals that the prepared phosphorus models are simple physical variants of $SrCO_3$, $CaCO_3$, and $NH_4H_2PO_4$. It has been observed that there are no peaks originating from the raw material for the obtained material. Also, the pattern (Figure 1) was very similar to that recently reported (13).

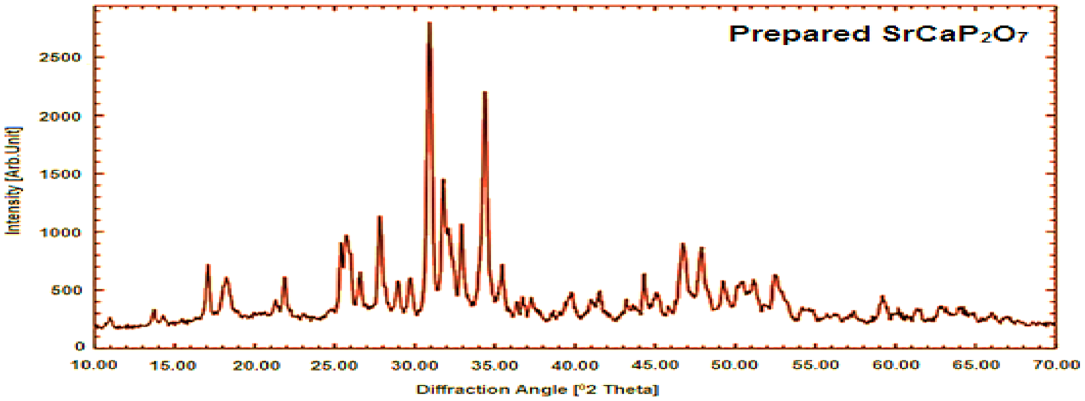

Figure 2: The XRD pattern of the $SrCaP_2O_7$ phosphor

3.2 SEM (Scanning Electron Microscope) characterization of $SrCaP_2O_7$ phosphor

The morphology of the products was examined with a scanning electron microscope (SEM, JEOL 6380A) and the distinctive morphological images are shown in Figure 3.SEM photographs of $SrCaP_2O_7$ phosphors clearly show that in submicrometer varieties the grains are irregularly shaped with a typical crystallite size. Particles are tripartite when they occur. The size of the phosphors prepared at 700^0C is about 6-10 nm. The extraordinary dispersion groove of the particles seems to start from the intense heat that interferes with the aggregation of the particles. Although the particles are attached to each other by necking, they can be simply crushed to obtain separate particles. Most of the particles were round surfaces and a few micrometers in size. The sample was subjected to sintered configurations in which certain particles would probably be barely noticeable. All these physiognomies were strategic in allowing them to be presented as phosphor powders for coating in the manufacture of White Light Emitting Diodes (W-LEDs) [14].

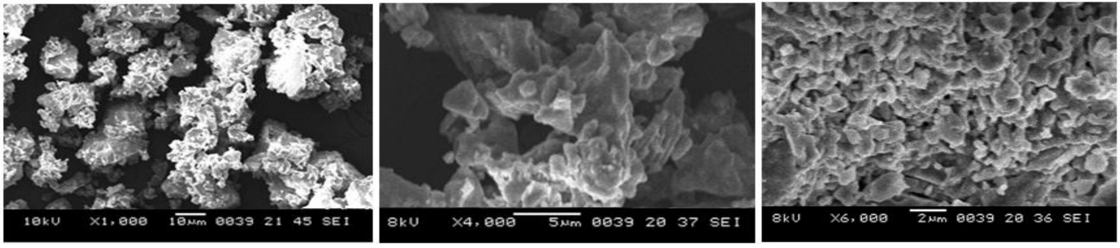

Figure 3: SEM micrograph of the $SrCaP_2O_7$ phosphor

3.3 Photoluminescence characterization:

3.3.1 Photoluminescence properties of Sm^{3+} activated $SrCaP_2O_7$ phosphor:

The synthesized Sm^{3+} doped $SrCaP_2O_7$ phosphors were also studied for their photoluminescence characterization with 0.1%, 0.3, 0.5 and 1 mol Sm concentrations. Figure

4 shows the excitation spectra of $SrCaP_2O_7:Sm^{3+}$ phosphor monitored at emission wavelengths of 564 nm. Excitation peaks are observed in the wavelength region from 300 nm to 450 nm. Found at 362, 376, 422 nm corresponding to $^6H_{5/2}\rightarrow{}^4D_{3/2}$, $^6H_{5/2}\rightarrow{}^4D_{1/2}$, $H_{5/2}\rightarrow{}^4L_{5/2}$, $^6H_{5/2}\rightarrow{}^4F_{7/2}$ several excitation peaks are attributed to f–f forbidden transitions of Sm^{3+}. $^6H_{5/2}\rightarrow$ (^6P, ^4P) $_{5/2}$, respectively. The intensity of the f-f transition at 422 nm was found to be higher than the other transitions; therefore this transition is chosen for measurement of the emission spectrum of $SrCaP_2O_7:Sm^{3+}$phosphoruses. This also indicates that $SrCaP_2O_7:Sm^{3+}$phosphors can be excited efficiently by near-UV (422 nm) light emitting diodes [15].

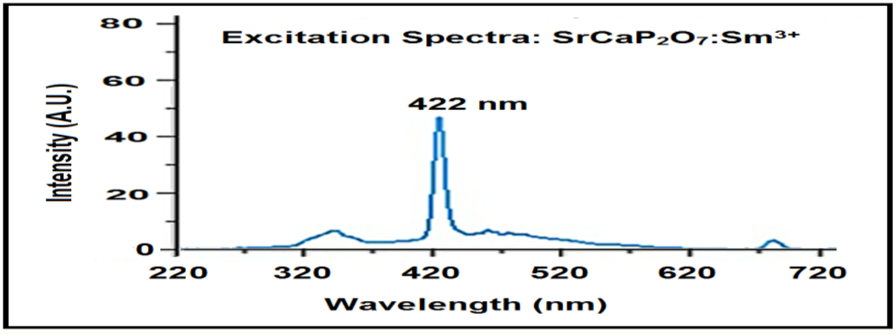

Figure 4: Excitation spectrum of $SrCaP_2O_7$: Sm^{3+} phosphor

The peak band of the emission spectrum ranges from 550 to 650 nm, where the main peaks are located at 564 nm, 602 nm and 647 nm as shown in figure 5, these emission peaks correspond to transitions $^4G_{5/2}\rightarrow{}^6H_{5/2}$, $^4G_{5/2}\rightarrow{}^6H_{7/2}$ and peaks, the peak at 564 nm is the dominant one. The peak at 564 nm is due to the magnetic dipole transition [16]. The peak at 602 nm is due to partly magnetic and partly electric dipole transition, while 602 nm peaks are sensitive to the crystal field environment and electric dipole is allowed. Since the peak at 564 nm is dominant, it indicates that Sm^{3+} ions are in the symmetric region in the host [17]. Figure 6 indicates energy emissions and transition of Sm^{3+} ions.

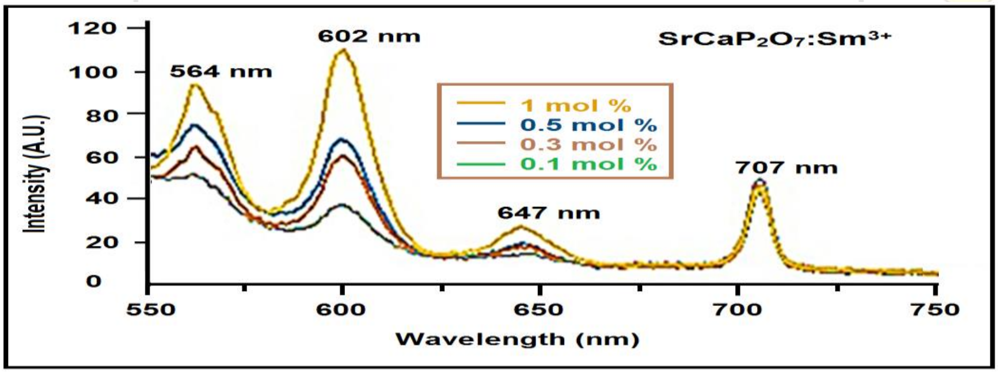

Figure 5: Emissions spectrum of $SrCaP_2O_7:Sm^{3+}$ phosphor

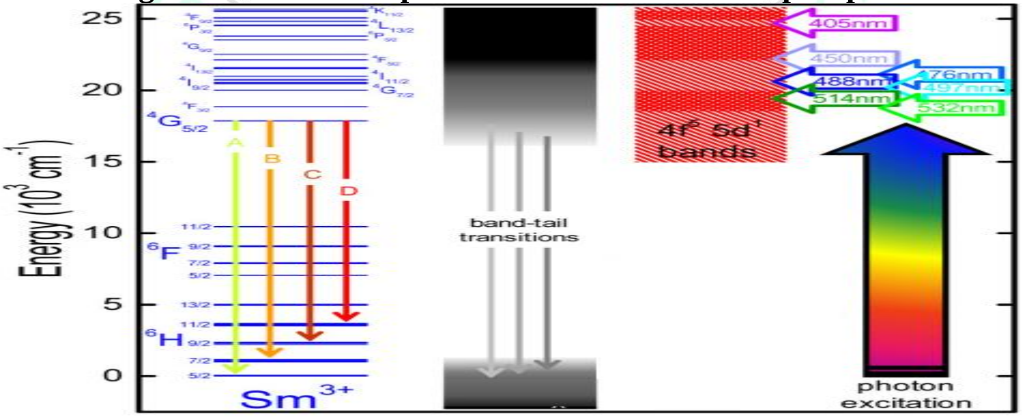

Figure 6: Energy emissions and transition of Sm^{3+} ions Adapted from A. Zanatta, "Coexistence of Sm^{3+} and Sm^{2+} ions in amorphous SiOx: origin, main light emission lines and excitation-recombination mechanisms," Opt. Mater. Express 6, 2108-2117 (2016) [18

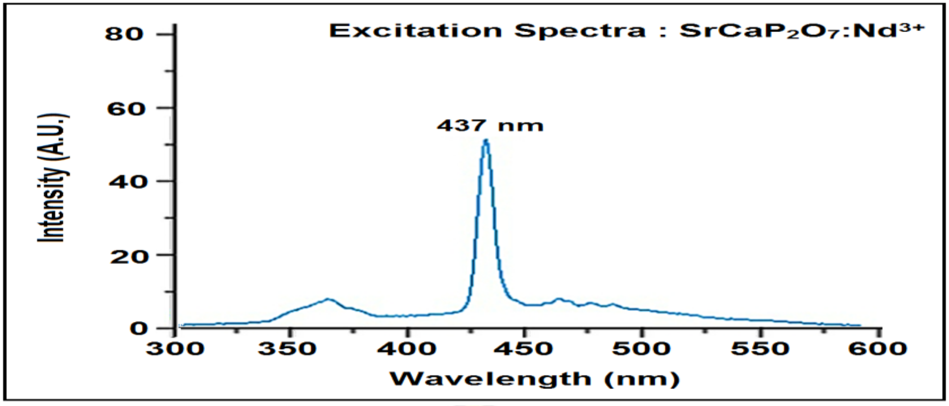

Figure 7: Excitation spectrum of $SrCaP_2O_7$: Nd^{3+} phosphor

3.3.2: Photoluminescence properties of Nd^{3+} activated $SrCaP_2O_7$ phosphor:

Figure 7 shows the excitation spectrum of $SrCaP_2O_7:Nd^{3+}$ while monitoring the orange emission at around 800-1000 nm, revealing that the doped samples have a sharp UV excitation peak located at 437nm. Excitation at 437 nm is attributed to band-to-band transitions. While monitoring the excitation wavelength at 437 nm, the results showed an emission bands between 700-1000 nm. Figure 8 shows the emission spectra of the Nd^{3+} activated $SrCaP_2O_7$ phosphor concentration series monitored at 437 nm excitation. The perceived spectra encompass characteristic bands corresponding to transitions $^4F_{3/2}$ level: $^4F_{3/2}$ –$^4I_{9/2}$ (850–990 nm), $^4F_{3/2}$ –$^4I_{11/2}$ (1020–1200 nm) and $^4F_{3/2}$ –$^4I_{13/2}$ (1300–1465 nm). Each emission transition of Nd^{3+} activated phosphors habitually consists of several well-resolved narrow lines corresponding to the Stark division between excited and ground levels due to the crystal field. Conversely, unsettled broad emission peaks were spotted in the case of $SrCaP_2O_7:Nd^{3+}$ phosphors. This statistic can be clarified as follows: The multisite circulation of Nd^{3+} ions in the spinel structure clues to line widening. The emission spectra governs $^4F_{3/2}$-$^4I_{11/2}$ transition positioned at 1075 nm. The wide-ranging band can be ascribed to the charge transfer transition (CT) between O^{2-} and Nd^{3+} ions. Extensive wavelength peaks resemble to intra-configurational f-f transitions Liu et al. [19] reported a broad emission peak centered amongst 700-1000 nm under UV excitation of 370 nm. They attributed the emissions to defect states such as interstitial oxygen [20].

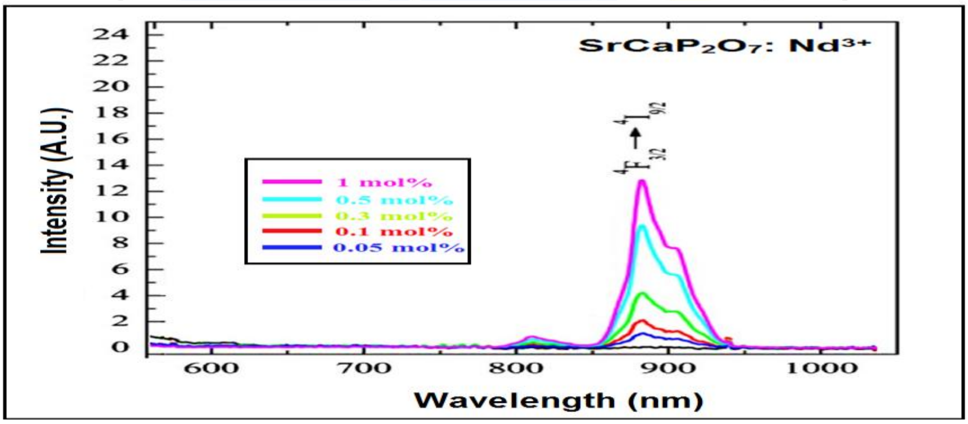

Figure 8: Emission spectrum of $SrCaP_2O_7$: Nd^{3+} phosphor

Fundamental concentration of this adaptation deliberated over the 800–1000 nm range as a function of Nd^{3+} concentration plotted in Fig. 8 to define the best doping concentration. There are twofold reasonable effects arising along with an intensification in doping concentration that move at the optimum value of the doping concentration: progression of radiative recombination and development in non-radiative degeneration ratio (energy transmission) [21]. As can be understood, the emission intensity is growing inadequately doped samples (0.05 and 0.5 mol %), but a further increase in Nd^{3+} doping ions results in decrease in intensity at 1mol% owing to concentration quenching effects. Optimum concentration of $Nd^{3+}$was found to be 0.5 mol.% in the $SrCaP_2O_7$ host. Figure 9 reveals the energy level diagram for Nd^{3+} ions in the present phosphor. The electric dipole transitions that produce emission wavelengths at approximately 800 nm are shown.

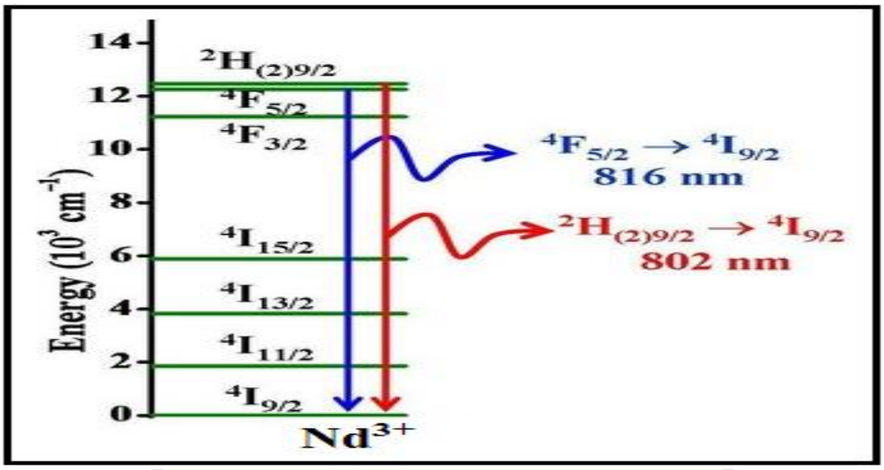

Figure 9: The energy level diagram for Nd^{3+} ions in the present phosphor

3.4 FTIR: Fourier transforms infrared structural behavior of $SrCaP_2O_7$

The room temperature FTIR spectrum in the range of 4600–400 cm^{-1} was recorded, and the FTIR spectra of the materials exhibited distinct multiple vibrational bands specifically in the four frequency regions (i.e. n_1 = 3954.09–3535.64 cm^{-1}, n_2 = 2500.66–2337.9 cm^{-1}, n_3 = 1811.79-1432.65 cm^{-1} and n_4 = 1249.65-412.58 cm^{-1}, See Fig. 10). Corbridge et al. [22] reports that, the P_2O_7 group (which can be written as O_3P-O-PO_3) can be defined as a combination of the vibrations of the PO_3 and P-O-P groups for vibration spectrum interpretation. The assignment of $P_2O_7^{4-}$ modes is carried out in terms of vibrations PO_3 and P-O-P. In these conditions, the infrared bands (Figure 10) for $SrCa(P_2O_7)$ are distributed in four different wave number ranges: 400–645, 720–750, 850–990 and 990–1400 cm^{-1}. Since the broad band between 1400 and 990 cm^{-1} resolved amongst many peaks, all vibrations of PO_3, PO_4 and P-O-P groups could be observed. The peak observed at 745 cm^{-1} can be attributed to the symmetrical stretching of the P-O-P bonds in the P_2O_7 group. As the degree of polymerization increases in [PO_4], the degree of multiplicity and fineness in the phosphate spectrum increases.

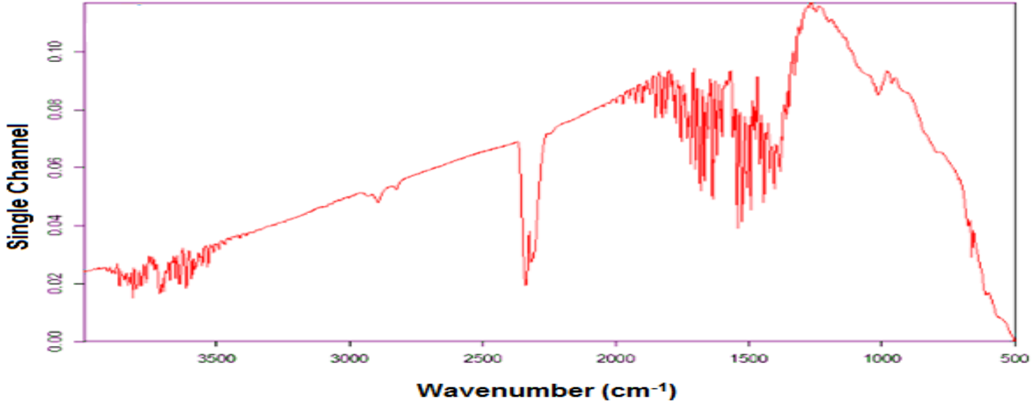

Figure 10 Fourier transforms infrared structural behavior of $SrCaP_2O_7$

Conclusion:

In this study, phosphate phosphors based on strontium calcium pyrophosphate ($SrCaP_2O_7$), activated with rare earth ions Sm^{3+},Nd^{3+}, prepared by solid state diffusion method and confirmed by XRD, were reported. An average crystallite size from the SEM study was found to be in the submicrometer range of 200-800 nm. PL spectroscopic characterizations of the prepared phosphors were performed using excitation and emission spectra. Photoluminescence spectra of rare earth activated $SrCaP_2O_7$ based phosphors show emission at 400-700 nm when excited between 200-400 nm.The results suggest that this phosphor may be useful for blue, red and orange light. The most efficient RE^{3+} concentration for maximum emission intensity is 1 mol%. All results show that the prepared phosphors could be potential phosphors for possible applications in solid state lighting and Light-emitting diodes (LED) field.

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