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Rare Earth Doped Nanophosphors: A Detailed Systematic Review of Recent Progress

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ABSTRACT:
Background: Rare Earth Doped Nanophosphors, with their distinct properties compared to bulk materials, have become a significant focus in nanotechnology research. Various synthesis methods, including solid-state reaction, sol-gel, and combustion, have been explored. However, challenges such as high processing temperatures, extended durations, and issues like luminescence degradation or agglomeration persist, highlighting the need for optimized approaches.
Purpose: This study systematically reviews the characteristics of nanophosphors synthesized using the combustion method, providing a comprehensive overview.
Methods: This review systematically analyzes nanophosphors synthesized via the combustion method. The methodology involves evaluating the effects of key synthesis parameters, such as fuel type, oxidizer flow rates, and precursor solution concentration, on particle characteristics. The combustion method's ability to provide a high-temperature environment and produce hollow-free nanophosphors is highlighted, offering insights into the tunability of size, morphology, and luminescence properties.
Results: The review examines the impact of dopant type, mole percentage, and codopants on the photoluminescence intensity of nanophosphors. It was observed that while dopant concentration does not alter the shape, morphology, or emission spectra, these properties can be enhanced by incorporating fluxes. Additionally, the dopant's mole percentage significantly influences the emission intensity of the phosphors.
Conclusions: This review analyzes over 45 papers from the last 15-20 years, focusing on rare-earth ion-doped nanophosphors synthesized using the combustion method. The study provides an in-depth examination of key characteristics such as particle size, crystalline size, surface morphology, and photoluminescence transitions.

Keywords: Rare Earth Doped Nanophosphors, Nanophosphors, Combustion Synthesis Method, Particle Size

1. Introduction

Extensive research has been conducted in the area of phosphors. Over the last few decades, there has been a growing interest in this field, leading to numerous investigations and advancements. Previously, phosphor was regarded merely as an element, but it is now recognized as a material that exhibits luminescent properties when exposed to light. The term 'phosphor' was first associated with luminescence in 1603. This journey began with a synthetic material when Vincenzo Cascariolo, an alchemist and cobbler, discovered a heavy crystalline stone with a glossy appearance at the foot of a volcano in Bologna, Italy. The sample was taken from the place known as Monte Paderno (Bologna). They heated a mixture of barium sulfate, $BaSO_4$ (in the form of barite, heavy spar) and coal. The residue that was obtained was in the form of powder and it showed a bluish glow in dark. Then Cascariolo realized that we can also store this glow by exposing that material (powder) to sunlight. That's why it was named "Sunstone" or 'emphlapis solaris' because the sun represents gold and in the beginning, the alchemists were expecting that it will turn the metals into gold.

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It was also recognized as 'Bolgonian stone'. These materials were lately called 'Phosphors'. Thus phosphor is also known as a light-bearer that emits light in dark [1]. After that in 1817, J. F. John also came up with one more sulphide-based phosphor-SrS [2]. Similarly, in 1866, a French chemist named Theodore Sidot successfully made a sulfide based phosphor i.e. ZnS [3]. Although, it was prepared accidentally, but was a prototype phosphor for a TV tube. Later it comes out to be one of the most important approaches in this field. V. Cascariolo was the person who gave us the first phosphor that was Barium Sulphide. However it was not considered as the first commercially available phosphor. It was in 1870 when V. Cascariolo gave its phosphor named as "Balmanian paint" that was further preparation of 'Barium Sulphide' (BaS) [4]. Earlier, the science behind such behavior of Bologna stone was a complete mystery. Since then efforts have been made to resolve it. A large variety of materials have been synthesized by adding different metals as impurities (also known as dopant/activators). In 1888, it was Eilhard Wiedemann who suggested that there are more types of phosphor present out there. He mentioned that these types are divided into proper classes based on the type of excitation followed [5]. In late 1920's and 1930's, P. W. Pohl and co-workers in Germany investigated alkali halide phosphors [6]. They also prepared a single crystal Phosphor. In these Tl-ion is used as a dopant. After this collaborative work with F. Seitz, the team of P. W. Pohl also succeeded in the establishment of a configuration model of luminescence centres. They also organized a foundation for modern luminescence physics. After world war two, optical spectroscopy helped in evolving the research of phosphor and solid-state luminescence. It resulted in the development of coloured TV in 1960.

The researchers have synthesized numerous phosphors using abundant experimental techniques including sol-gel, hydrothermal, microwave, solid-state reaction and solution combustion. In general, the rare-earth doped phosphors are mainly synthesized by the solid-state reaction at high temperature. However, this method requires a high processing temperature, a long processing time, repeated milling and washing with chemicals. These processes tend to degrade the luminescence property of the particles and yield irregularly shaped particles. Sol-gel method has also been employed to synthesize nanophosphors by many researchers. The as-prepared powders obtained from the sol-gel method have low crystallinity and often require post-treatment at high temperature, which results in severe agglomerations. Combustion synthesis is a promising particle preparation method because it can employ a wide range of precursors for synthesis of a broad spectrum of functional nanoparticles. The use of combustion can avoid hollowness and provide the high temperature environment which is favourable to phosphor synthesis. The flame temperature and particle residence time, which are very important parameters determining particle characteristics, can be easily controlled by varying fuel and oxidizer flow rates. Moreover, the particle size can be controlled by varying precursor solution concentration and multi-component particles can also be obtained by adding different salts into the solution. Therefore, a review of the characteristic properties of nanophosphors prepared using combustion method is conducted in the present study. In this review, we have revealed various characteristics of nanophosphors i.e. particle size, morphology, photoluminescence properties as well as their applications in various fields.

2. Methodology and Materials

Combustion Synthesis Method is a method which is used as a substitute synthetic route in phosphors activated with europium, cerium etc. The use of combustion technique can help in avoiding hollowness and it provides high temperature environment which is favourable for the synthesis of phosphor. It is an economical method as it provides uniform, crystalline, oxide nanoparticles in comparison to the time-consuming techniques like solid-state reaction and sol-gel processing [7, 8]. This method was discovered in 1988 by Patil and co-workers [9]. It is a self propagating high temperature synthetic technique and an efficient and energy saving approach. It is an economical method for the synthesis of very closely agglomerated, multicomponent oxide ceramic powder [10]. It is a promising particle preparation method because it can employ a

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wide range of precursor for the synthesis of broad spectrum of functional nanoparticles. Precursors used in the combustion synthesis technique are nitrates and acetate salts. An appropriate quantity of organic 'fuel' such as urea, glycine, ethylene glycol etc. in an appropriate stoichiometric ratio is added to the aqueous solution of the salts. Further heating of mixture on hot plate is done to evaporate the water in order to obtain a gel. The gel is again heated in a pre-heated oven (300-500 Celsius) which converts it into fluffy powdered form. The powder is then reheated at 600-800 Celsius in air to burn all the organic residues. It further consists of two modes:

2.1. Self-propagating High Temperature Synthesis (SHS)

It was originally discovered by A. G. Merzhanov in 1967. In this case the media is locally preheated using some external source to its ignition temperature. It is the temperature at which the reaction gets initiated in this layer. Further this preheated layer ignites next cold layer and thus combustion front self propagates along the reactive mixture resulting in the formation of desired solid product.

2.2. Volume Combustion Synthesis Mode

Here the entire reactive layers are uniformly heated instead of preheating by some external source. The source provides ignition temperature and the reaction starts at each point of the media uniformally leading to the production of some valuable materials. Sometimes fuels and oxidizers are also required for the reaction to be carried out. In this synthesis metal nitrides acts as oxidizer whereas nitrogenic compounds such as urea and glycine are used as fuels. Fuel and oxidizer are also required for the combustion reaction. In combustion synthesis of the oxides, fuels used are nitrogen containing organic compounds and metal nitrates acts as oxidizer. For calculating the proper ratio of fuels and metal nitrates, propellant chemistry is used.

2.3. Combustion synthesis of phosphors using solution combustion

Solution combustion synthesis (SCS) is a fast process, which allows efficient production of variety of nano-size materials. This method involves a self-sustained reaction in uniform solution of diverse oxidizer (e.g., metal nitrates) and fuels (e.g. urea. carbohydrazide, semicarbazide). SCS take place as either volume or layer-by-layer proliferating combustion method, as the two way depend on the nature of the precursors, as well as on conditions used for the process organization. The method capitulates nano-size oxide materials along with homogeneous doping of trace amounts of rare-earth impurity ions in a single step. The most recent improvements in SCS method are discussed based on the materials applications. The production of nanophosphors in recent years attracted attention of various researchers in the field of combustion synthesis. A lot of nanophosphors based materials prepared by SCS [11, 12, 13] $Y_2SiO_5 : Ce, Lu_2SiO_5 : Ce, Gd_2SiO_5 : Ce$. Fuel used for these synthesis are Hexamine and Urea. It is clear that urea persists to be the ideal fuel for phosphor material production. It provides highly pure, crystalline and homogeneous powder. It involves low temperature, which makes it more efficient in comparison to the conventional method [14].

2.4. Benefits of the Combustion Synthesis Method

I No external sources will be required once ignition occurs.

II It is an energy efficient technique and requires low energy.

III It provides ultra fine powder in short interval of time.

IV It provides high purity products.

V It is a rapid process.

VI It provides molecular level of mixing and high degree of homogeneity.

VII It is quite a simple process and also requires low cost.

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Table 1: The collected data for various characteristic features of rare earth doped nanophosphors using the Combustion Method.

Dopant	**Co dopant**	**Host**	**XRD Analysis**	**TEM Analysis**	**PL Analysisr**		**SEM Analysis**	**Remarks**	**Ref No.**
			Particle Size (nm)		**Transitions**	**λ(nm)**	**Morphology Study**		
Eu^{3+}		$MgGdAl_3O_7$, $CaGdAl_3O_7$, $SrGdAl_3O_7$, $BaGdAl_3O_7$	19.86, 40.29, 19.57, 23.22	18.74, 30.87, 19.18, 14.52	$5D_0 \rightarrow 7F_2$, $5D_0 \rightarrow 7F_2$, $5D_0 \rightarrow 7F_2$, $5D_0 \rightarrow 7F_2$,	608–614 (red)	spherical, Spherical, Spherical, Spherical,	Optimal concentration of Eu^{3+} ions is 0.03 mol%. Application: used in WLEDs and modern display devices.	[15]
		$BiVO_4$	23	1-2 μm	$5D_0 \rightarrow 7F_2$,	615 (red)	spheroidal	Intensity = 1 mol %	[16]
		Gd_2O_3	48-76	60-155	$5D_0 \rightarrow 7F_2$,	612 (red)	Cubic	Intensity = 8 mol%. Temp = 800 °C.	[17]
		$BaGd_2ZnO_5$	89-98	80-90	$5D_0 \rightarrow 7F_2$,	628 (intense red)	Spherical	PL Spectra is at 1100 C. Optimum concentration is 4 mol %.	[18]
		$CaWO_4$		20-200	$5D_0 \rightarrow 7F_2$	614 (red)	Irregular	Optimum concentration for highest luminescence is at 20 mol %.It is an ethylene glycol medium.	[19]
		Sr_2SiO_4	45-60	50-60	$5D_0 \rightarrow 7F_2$	612 (red)	Irregular	Maximum PL intensity at 4 mol %.Used in radiation dosimetry.Low temp SCT.	[20]
		$LaAlO_3$	70	70	$5D_0 \rightarrow 7F_2$	618 (red)	Circular	Maximum intensity at 5 mol %. Application-WLED's and solid state lighting.	[21]
		$CaYAl_3O_7$ $SrYAl_3O_7$ $MgYAl_3O_7$ $BaYAl_3O_7$	10.61 23.73 19.57 28.09	14.81 25.29 25.15 33.11	$8S_{7/2} \rightarrow 6I_{7/2}$	254 (red)	Highly agglomerated	Temp. = 600 C, Intensity = 0.03 mol % Variety of display applications.	[22]

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(Table continued..)

Dopant	Co dopant	Host	XRD Analysis	TEM Analysis	PL Analysisr		SEM Analysis	Remarks	Ref No.
			Particle Size (nm)		Transitions	λ(nm)	Morphology Study		
		$SrTiO_3$	25-30	32	$5D_0 \rightarrow 7F_2$	614	Irregular agglomerated	Various fluxes are added. NH4F is appropriate one which reduces the forming temp, improve morphology, enhance luminescence intensity.	[23]
		Gd_3PO_7	40	40	$5D_0 \rightarrow 7F_2$	614	Spherical	Maximum intensity at room temp of 304 K . Optical temp. for this material as red phosphor is 304 K , about room temp. facile combustion method.	[24]
		MgY_2O_4 CaY_2O_4 SrY_2O_4	41.03 16.74 19.35	10-25	$5D_0 \rightarrow 7F_2$ $5D_0 \rightarrow 7F_2$ $5D_0 \rightarrow 7F_2$	612 (red) 612 612	Spherical agglomerated	Emission intensity is maximum. Application- used as solid state lightening.	[25]
Eu^{2+}	Dy^{3+}	$SrAl_2O_4$	30.38	25-35	$8H_{3/2} \rightarrow 8S_{7/2}$,	510 (green)	Mostly spherical	Used as fuel. Used rare earth ions as co-dopants. Application in solar cells and lighting technology.	[26]
		BaB_8O_{13}	14.13		$5D_1 \rightarrow 4F_7$,	400 (Blue)	Foamy and agglomerate	SCT	[27]
		$SrAl_4O_7$	17.86	10-40	$5D_1 \rightarrow 4F_7$,	504 (green)	Flake like morphology	Rapid gel CT Co-doping improved the phosphorescence decay lifetime. Used in display devices.	[28]
Dy^{3+}		$BaLaAlO_4$	49	30.2	$4F_{9/2} \rightarrow 6H_{15/2}$,	481 (Blue)	Orthorhombic Agglomerated	Maximum intensity= 3.0 mol	[29]
		$SrLa_2Al_2O_7$	45	45-52	$4F_{9/2} \rightarrow 6H_{13/2}$,	575 (Yellow)	Spherical	Application in WLED's.	[30]

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(Table continued..)

Dopant	Co dopant	Host	XRD Analysis	TEM Analysis	PL Analysisr		SEM Analysis	Remarks	Ref No.
			Particle Size (nm)		Transitions	λ(nm)	Morphology Study		
		Sr_2SiO_4	37	17	$4F_{9/2} \rightarrow 6H_{13/2}$,	478(Blue)	Irregular spherical	Intensity = 1 molWhite light emission rises from dosimetry in the irradiation of food products.	[31]
		$Ca_2MgSi_2O_7$	46	50	$4F_{9/2} \rightarrow 6H_{15/2}$,	480(Blue)	Dumb Bell	Energy band gap =3.52-3.68 e V. Used in WLED's.	[32]
Tb^{3+}		$CaSiO_3$	68.50		$5D_4 \rightarrow 7F_5$,	555 (green)	Hexagonal	Maximum intensity = 1-10 mol % Applications-Used in cheap green organic salts.	[33]
		LaOF	25-45		$5D_4 \rightarrow 7F_j$,	545 (green)	Irregular Shape	Intensity = 1-9 mol %. PL intensity increase up to 3% and then decreases. Application-Fingerprint identification	[34]
		$LaAlO_3$	60	60	$5D_4 \rightarrow 7F_5$,	542(Green)	Spherical	Temp = 800 C Intensity depends on Tb concentration. Maximum intensity at 10 Used as glycine acid in fuel.	[35]
Er^{3+}	Yb^{3+}	$SrGdAlO_4$	49.5	49-55	$4S_{3/2} \rightarrow 4I_{15/2}$,	549 (Briight green)	Spherical	Optimal concentration = 4 mol %.Used in WLED's.	[36]
		Gd_2O_3	38-49	60-155	$4F_{9/2} \rightarrow 4I_{15/2}$,	652 (red)	Cubic phase	Temp = 800 C.Intensity = 8 mol %.	[37]
Ce^{3+}		$Y_3Al_5O_{12}$	a = 1.2009	40	5D $\rightarrow$ 4F	560 (yellow)	Cubic	Intensity = 6-7 mol%. Application: candidate for generating WLEDs when coupled to blue LEDs.	[38]

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(Table continued..)

Dopant	Co dopant	Host	XRD Analysis	TEM Analysis	PL Analysisr		SEM Analysis	Remarks	Ref No.
			Particle Size (nm)		Transitions	λ(nm)	Morphology Study		
		$LaAlO_3$	45-49	35-40	$5D \rightarrow 2F_{7/2}$,	330 (Bluish-Green)	Highly agglomerated	Maximum PL at 1.5 mol %. Application - suitable for the fabrication of display and optical devices.	[39]
		$CdSiO_3$	30-40		$2D \rightarrow 2F_{5/2}$,	450-700 (yellow)	Monoclinic	Intensity = 1-11 mol % Used as oxalyl di-hydrazine fuel. Low-temperature solution combustion method.	[40]
Sm^{3+}		$CaTiO_3$	20	10-18	$4G_{5/2} \rightarrow 6H_{7/2}$	601-611 (orange-red)	Porous	Low temp. solution combustion method. Intensity = 1-11 mol % Used in WLED's.	[41]
		Y_2SiO_5	10-50	10-50	$4G_{5/2} \rightarrow 6H_{7/2}$	600-613 (orange-red)	Irregular	Intensity = 1-9 mol%.Used in WLED's.	[42]
Mn		(Ba,Sr) $MgAl_{10}O_{17}$	78	Dia-80	$4T_{1G} \rightarrow 6A_{1G}$	518 (green)	Spherical	Application-Plasma display panels, 3D displays and mercury free fluorescent lamps.	[43]
SM_1 SM_2		$SrMoO_4$, $SrMoO_4$	19 27	10-40 15-50	$3T_{1,2} \rightarrow 1A_1$	397 (blue) 523(green)	tetragonal	Highly porous Study the influence of different molybdenum sources.	[44]

Therefore, it is a promising phosphor synthesis technique as it can employ a wide range of precursor for the synthesis of broad spectrum of functional nanoparticles.

3. Results and Discussion

In this study, we reviewed the literature from the past 15 years to explore the characteristic properties of nanophosphors synthesized using the combustion synthesis technique. The reviewed literature primarily focuses on rare-earth ion-doped nanophosphors prepared through this method. The collected data highlights various characteristic features of these nanophosphors, which are summarized in Table 1. This includes details on particle size, crystalline size, surface morphology, and photoluminescence transitions. The data presented in Table 1 has been gathered using specific characterization techniques to determine the properties of the phosphors.

i X-Ray Diffraction (XRD)

ii Transmission Electron Microscopy (TEM)

iii Scanning Electron Microscopy (SEM)

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iv Photoluminescence (PL)

After a detailed review of the papers, we observed certain consistent patterns, few are listed here:

i While combustion synthesis can produce materials of various sizes detectable by XRD, the average crystallite size of nanophosphors, as calculated using the Debye-Scherrer formula, is predominantly found to be below approximately 50 nm.

ii For XRD studies, the average crystallite size is calculated using the Debye-Scherrer formula. The formula is given as:

$$D = K\lambda/(\beta\cos\Theta) \quad (1)$$

For cubic crystal structure, K =0.941, λ is wavelength of X-ray. β and θ are the FWHM (radians) and diffraction angle of the observed peak respectively. In Ref. [21], the XRD patterns of the $La_{1-x}AlO_3 : xEu$ (x=0.03, 0.09, 0.15 and 0.21) phosphor which is synthesized by solution combustion synthesis technique is shown. It gives a pure rhombohedral structure. XRD peaks are given for different mol % concentration of the europium ion. It has been observed that change in mol concentration of dopant i.e. Eu^{3+} has almost no effect on the phase composition. Even on increasing the concentration of Eu^{3+} ion to 21% no impurity phases are observed. On applying Debye Sherrers formula to the diffraction peak (110) of the $LaAlO_3$ crystal, the average crystallite size comes out to be 70nm [21].

iii The particle size observed using TEM studies is also found in good agreement with XRD data in some cases. TEM also provides details about the shape, crystallinity and some of the luminescence properties of the material. Ref. [45] displays the TEM image of $LaAlO_3 : Eu^{3+}$ nanophosphors. In case of TEM studies, the data for particle size of the nanophosphors is collected. TEM studies are carried out to understand the crystalline characteristics of the nanoparticles. The particles observed are mostly in spherical shape having their particle size ranging between 20-50nm. It also reveals about the formation of high crystalline nanophosphors [45].

iv It is observed that by increasing the annealing temperature, we can improve the crystallinity of the material.

v The morphological details are taken using SEM technique. In most the cases we have observed nanophosphors showing irregularity in shape, high porosity and agglomeration as escape of large amount of gases takes place during synthesis process. The surface morphology of pure and $Eu^{3+}(1-9mol\%)$ doped $LaAlO_3$ nanophosphors prepared via a combustion method is studied using SEM and is shown in Ref. [45]. It is clearly evident that, the powders show highly porous, agglomerates with an irregular morphology, large voids, cracks, pores and shapes. This type of morphology is due to the escape of a large volume of gas during combustion process. Further, the dopant concentration does not influence the morphology of the sample [45].

vi The most prominent emission takes place for $5D \rightarrow 4F$ transition within 600-630nm range resulting in highly intense emission in red colour region along with some other colours. Ref. [21] illustrates the emission spectra under 394nm ($^7F_0 \rightarrow {}^5L_6$) and 464nm ($^7F_0 \rightarrow {}^5D_2$) excitations for $La_{1-x}AlO_3 : xEu^{3+}$ nanophosphor. One should note that the profile of the emission spectra are similar, also that the emission spectra of red light at 618nm is the predominant one with strongest intensity. It was also observed that the increase in mole % concentration of the activator/dopant increases the intensities of transitions upto 12% only. On surpassing this value the intensity starts decreasing gradually, thus we can get maximum intensity emission of red only upto 12% for Eu^{3+} ion dopant. Under different excitations the quenching value for PL also

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changes. It is also observed for 394/464nm excitations by using CIE chromatography diagram gives same result.

vii Effect of codopants is also seen on photoluminescence intensity, but no effect is observed on the shape and profile of emission spectra.

viii The results are mostly calculated by varying the mole percentage of dopant and the optimum value of dopant mole percentage is selected from the photoluminescence intensity values. After the optimal concentration of dopant ions, concentration quenching phenomena occurs.

ix It has been found that the concentration of dopant doesn't effect the particle size, crystallinity and morphology of the material.

x Lanthanide-activated nanophosphors, especially ones with aluminate host lattice shows excellent luminescent applications.

xi The localised environment of the dopant/activator also plays a vital role in providing persistent luminescence.

xii Addition of flux shifts the CIE coordinates from orange-red to white region along with enhancing the PL properties.

xiii It is also evident from the results that with dopant Eu^{3+}, the prominent emission colour is red which is irrespective of the host material.

xiv Moreover, with Dy^{3+} activator, white light emitting phosphors are formed and they having practical application in white light emitting diodes (WLED).

4. Findings and Conclusion

This study presents a comprehensive review of over 50 papers published in the last 15-20 years, focusing on rare-earth ion-doped nanophosphors synthesized via the combustion synthesis method. The reviewed literature extensively covers key characteristics such as particle size, crystallite size, surface morphology, and photoluminescence transitions. Analysis of the studies reveals a consistent trend where the average crystallite size, determined using the Debye-Scherrer formula, is predominantly below approximately $\approx 50nm$. Furthermore, the particle sizes obtained through Transmission Electron Microscopy (TEM) are generally consistent with those derived from X-ray Diffraction (XRD) analysis. Notably, for phosphors doped with Eu^{3+} ions, a prominent red emission is observed, regardless of the host material, whileDy^{3+}-doped phosphors exhibit white light emission, making them suitable for applications in white light-emitting diodes (WLEDs). This review consolidates these findings, offering valuable insights and a foundation for future experimental investigations in the field of nanophosphor research.

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