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Original Article

Photoluminescence in Synthetic Neighborite ($NaMgF_3$) halophosphor by doping Ce^{3+} and Eu^{3+} rare earths

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Abstract

This research discusses the Crystal structure, different synthesis routes and luminescence characteristics of halophosphor synthetic mineral $NaMgF_3$ (Neighborite) by doping rare earths (Ce, and Eu). Neighborite was synthesized by three routes, Viz. wet chemical synthesis (WCS), Solid state diffusion (SSD) and centrifuge route (CS). XRD of $NaMgF_3$ prepared by WCS, SSD and CS routes were matched with the standard ICSD 075291. Photoluminescence (PL) was observed in Pure $NaMgF_3$ in ultraviolet (UV) region at 388 nm. Self-luminescence in the material shows presence of some kinds of defects or electronic centers. $NaMgF_3$:Ce^{3+} shows PL emission in UV region and $NaMgF_3$: Eu^{3+} shows emission in Orange-red region. Maximum intensity in PL is observed when halophosphor is synthesized by WCS.

Keywords: *Halophosphor;* wet chemical synthesis; Solid state diffusion; centrifuge route *and photoluminescence.*

1. Introduction

The fluoride materials tend to be soft, brittle, easily soluble in water, and possess medium to high melting points. They are poor conductors of heat and electricity when in solid state but good conductors in molten state. Ions are liberated in molten state then transport charges. Fluoride compounds have large band gaps (9-10 eV) and these are known to be stiff lattices and are of significant interest in the scientific world because of their wide range of applications moreover doping of rare earth ions in fluorides produces efficient emission in ultra violet and in visible region leading these materials for important applications in the world of lighting. Fluorides doped with rare earths are very attractive materials for optical applications since they have very wide transparency in spectral range from VUV to far IR, weak crystal field and small refractive index. Due to low phonon frequencies ($\hbar\omega_{max} < 450\ cm^{-1}$) the excited states of lanthanides embedded into fluoride hosts exhibit long lifetimes, which leads to a small probability of non-radiative transitions and the number of luminescent levels is usually larger than for oxide crystals doped with Ln^{3+} ions. They exhibit lower phonon energies than oxides, but often are completely air stable materials in contrast to chloride or bromide hosts. Several fluorides have been shown to possess properties desired of solid-state laser hosts and as phosphors for mercury-free fluorescent lamps and plasma display panels [1, 2].

Masson et al. had studied the spectroscopic properties of Ce^{3+} and Pr^{3+} in $KMgF_3$ [3]. A spectroscopic study was also performed on $KMgF_3$: Eu^{2+} [4], BaF_2: Ce [5] and LaF_3:Ce[6] which showed emission in ultraviolet range whose decay time is 20 ns which is useful for efficient scintillation properties. Phosphors of Na_3SO_4F had studied by Gedam et al [7] and studied PL excitation spectra of $NaMgSO_4F$:Ce^{3+} phosphor. $KMgSO_4F$ halosulfate fluoride phosphors have been synthesized and investigated by A. Poddar et.al. [8, 9, 10]. $K_3Ca_2(SO_4)_3F$ had been synthesized by a co-precipitation method PL, TL characteristics of this halophosphor had been studied by doping cerium [11, 12]. A similar compound $KMgCl_3$ phosphor had been had been studied [13, 14].

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2. Experimental:

Synthesis of Synthetic $NaMgF_3$ Halophosphor

There are number of conventional and non-conventional methods of synthesis of $NaMgF_3$. Different synthesis routes are reviewed, and then same complex halide $NaMgF_3$ involving as many as three components are prepared by three synthesis routes, Wet Chemical, Solid State Diffusion, Centrifuge Synthesis and their luminescence properties were compared. These synthesis routes are attractive because of their capacity to yield products at low temperature. The processes are safe, simple, and it requires very simple experimental set up.

2.1 Wet Chemical Synthesis (WCS)

This is one of the simplest synthesis techniques currently used, it is also called as one step synthesis [15, 16]. There are number of advantages of the wet chemical synthesis. It is low cost and called power saving synthesis technique. It requires low temperature for reaction and chemical reaction proceeds very swiftly in the liquid state in water. Wet chemical synthesis is an ideal technique used for producing fine, chemically homogeneous and pure, single phase powder and can be exploited to prepare phosphor with enhanced optical properties. This method is not suitable for synthesis of the complex oxide phosphors.

The method for synthesis of $NaMgF_3$ explored the fact that the use of chlorine ion as starting materials prevents hydrolysis [17]. For synthesizing the samples by WCS route, complex fluorides were prepared by dissolving the analytical reagent grade chlorides of the constituents ($MgCl_2$, NaCl) in the stoichiometric ratio in double distilled de-ionized water in a PTFE container separately then mixed together and NH_4F was added drop wise. Resulting solution was stirred for half an hour with the help of magnetic stirrer. This was confirmed that all the salts dissolved completely in water to get homogeneous solution and no un-dissolved constituents were left behind. The solution was then allowed to evaporate till the mixture becomes anhydrous. The evaporation was done at 80°C for 8 to 10 hours. The dried samples were then slowly cooled at room temperature (RT). The resultant polycrystalline mass was crushed to fine particle in a crucible. The powder of pure $NaMgF_3$ was obtained in this way. For adding the impurities of rare earths, Sulfate salt of Cerium or Europium were used to get $NaMgF_3$:Ce or $NaMgF_3$:Eu. The chemical reaction in all the components is given by

$$MgCl_2 + NaCl + 3NH_4F \rightarrow NaMgF_3 + 3HCl + 3NH_3$$
$$NaMgF_3 + (Ce_2SO_4)_3.H_2O \rightarrow NaMgF_3:Ce^{3+}$$
$$NaMgF_3 + Eu_2(SO_4)_3 \cdot 8\,H_2O \rightarrow NaMgF_3:Eu^{3+}$$

2.2 Solid State Diffusion method (SSD)

Solid-state diffusion is one of the most widely used methods for the synthesis of halide materials. Solid-solid reactions are simple to perform; the constituents are made to react through diffusion process. The temperature is just enough to have adequate diffusion to complete the reaction in laboratory time without melting the constituents.

To prepare $NaMgF_3$ by SSD route same stoichiometric ratio of starting compounds were taken, mixed and grounded homogeneously in agate mortar to get pure $NaMgF_3$. The mixture was heated to 100°C in a crucible for 1h, in order to allow water to evaporate. The mixture was crushed once again and then this powder was heated once again at 300° C for 24h, cooled overnight to room temperature thereby obtaining the white phosphor powder. For adding dopants of rare earths, Sulfate salt of Cerium, or Europium were used and they are added in the mixture at the time of grounding to get $NaMgF_3$:Ce or $NaMgF_3$:Eu.

2.3 Centrifuge Synthesis Route (CS)

This refers to the synthesis of phosphor powders by using non-aqueous liquid media, which can be inert or constitute a reactant, for example ammonia or ethanol. The morphology of powders made in liquid-phase reaction is in general, controlled by calcinations times and temperature. This method allows low-temperature synthesis of phosphor powders. High purity, sub-micrometer / nano isolated particles with a narrow size distribution that can be obtained in these reactions. When phosphors are prepared by conventional methods usually unwanted impurities, like OH^-, O_2^- may incorporate, which can be controlled in this method. Another method used in synthesizing samples is CS route**.** Same stoichiometric ratio of starting compounds were taken in the Teflon beaker and 20 ml ethanol is added then stirred the mixture for about 10-12 hours with the help of magnetic stirrer. The resulting solution is centrifuged for 3-4 min at 4500 rpm to separate the compound. The supernatant was discarded. The precipitate was suspended in 30 ml de-ionized water and centrifuged again for 2-3 times. The powder was obtained by removing ethanol, which was treated at 40°C for few hours to get fine white powder of pure $NaMgF_3$. For adding dopants of rare earths and transition metals, Sulphate salt of Cerium or Dysprosium or Europium were used and they are added in the mixture at the time of stirring to get $NaMgF_3$:Ce and $NaMgF_3$:Eu.

3. Results and Discussion

3.1 X-ray diffraction pattern (XRD) of $NaMgF_3$

Formation of the compound, $NaMgF_3$ prepared by three different routes was confirmed by taking the x-ray diffraction (XRD) that matched with the standard data available. XRD pattern was formed when X-rays are focused on a powder material. Bruker D8 Advance X-Ray Difractometer was used which is equipped with a sealed tube Cu-Kα x-ray source. In XRD, a collimated beam of X rays, with wavelength, λ~1A°, is incident on a specimen and is

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diffracted by the crystalline phases in the specimen according to Bragg's law ($\lambda = 2d\sin\theta$, where d is the spacing between atomic planes in the crystalline phase). The intensity of the diffracted X-rays is measured as a function of the diffraction angle 2θ and the specimen's orientation. This diffraction pattern is used to identify the specimen's crystalline phases and to measure its structural properties. The photographs of the XRD were taken.

Figure 1 shows XRD of $NaMgF_3$ prepared by WCS, SSD and CS routes. XRD patterns were matched with the standard ICSD 075291. The measure lines of the pattern are matching with the standard ICSD, which is a direct evidence of the formation of the desired compound.

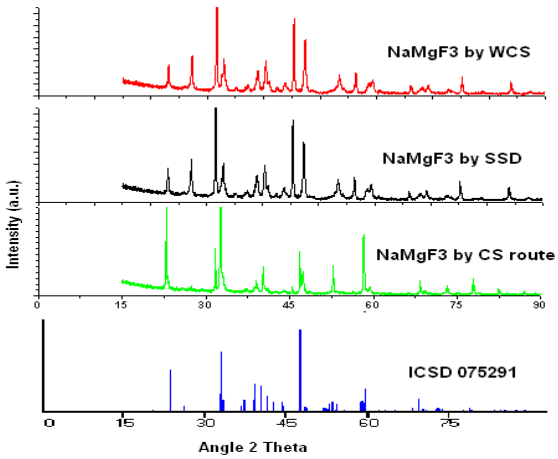

Figure 1: X-Ray diffraction pattern (XRD) of $NaMgF_3$ by WCS, SSD and CS route compared with standard XRD

3.2 Luminescence in pure $NaMgF_3$

The photoluminescence (PL) emission spectra of the samples were recorded using Fluorescence spectrometer (Shimatzu RF-5301) in the range 220–700 nm with spectral slit widths of 1.5 nm. The same amount of sample was used in each case. The Spectrofluorophotometer irradiates a sample with excitation light and measures the fluorescence emitted from the irradiated sample to perform a qualitative or quantitative analysis. Spectrofluorophotometer consists of monochromators (one on the excitation and other on the emission side), a light source, two detectors (one for measurements and the other for monitoring), a sample holder, a data processor and graphic plotter. As far as monocromators are concerned, the excitation monochromator incorporates a diffraction grating with a larger aperture to collect the largest possible amount of light. Emission monochromator has a diffraction grating whose size is the same as that of the excitation monochromator to collect the greatest possible amount of light. Xenon lamp of very high emission intensity and an uninterrupted radiation spectrum is used as a light source. The non-uniformity in the radiation spectrum of the Xenon lamp and in the spectral sensitivity characteristics of the photomultiplier tube causes distortion in the spectrum. To overcome these factors, the photomultiplier tube was used in the instrument.

The photoluminescence (PL) properties of pure $NaMgF_3$ obtained by WCS, SSD and CS route are examined. Figure 2 and Figure 3 show the excitation and emission spectra respectively. The excitation spectrum shows peak at 358nm and there is emission band with maximum at 388 nm. Since there are no activated ions present, so the emission has to be caused by some kinds of defects or electronic centers [18-19], showing its self-luminescence.

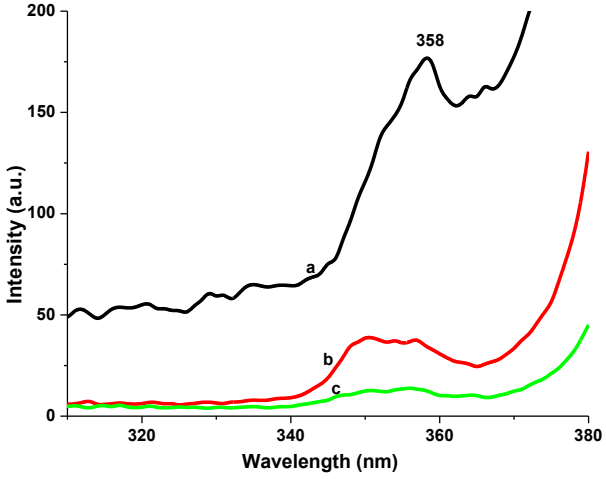

Figure 2: Excitation spectra of pure $NaMgF_3$ monitored at λ_{em}=389nm synthesized by a) WCS,b) SSD,C) CS

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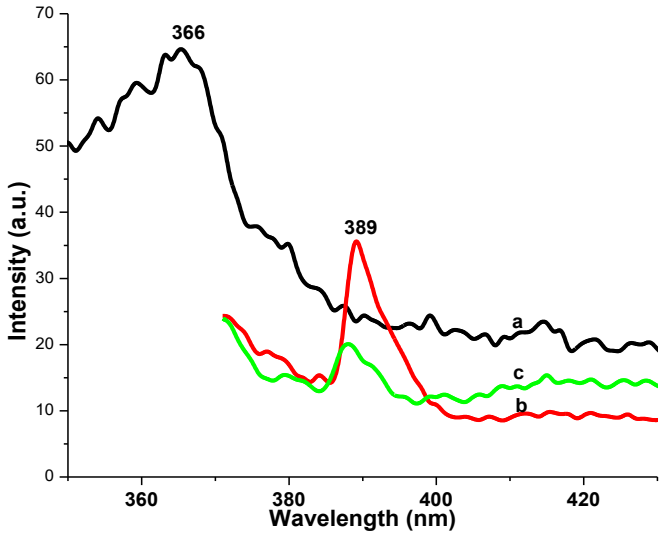

Figure 3: Emission spectra of pure $NaMgF_3$ monitored at λ_{ex} = 358 nm synthesized by a)WCS,b)SSD,C)CS

3.4 Luminescence in $NaMgF_3$:Ce^{3+}

Figure 4 shows the excitation spectrum of 2 m% of Ce^{3+} in $NaMgF_3$. The excitation bands are broad and remain at the same position, 286 nm when synthesized by WCS and SSD route and it was at 281nm when synthesized by CS route. All emission spectra are monitored at excitation wavelength of 286nm showing emission at 327nm (UV) and some amount of emission was observed in red at 642nm shown in figure 5 synthesized by WCS, b) SSD and c) CS routes. Literature survey on many Ce^{3+} doped inorganic host lattice, said that Ce^{3+} emission is mainly observed as in double hump peak or broad band emission, in ultraviolet to visible regions of the spectra that depends on host lattice. Masson et al had reported values for emission and excitation bands of Ce^{3+} in $NaMgF_3$ as 303 nm and 245nm at room temperature prepared by melting zone technique. In fluorides, For Ce^{3+} emission it is quite common to observe the longer wavelength band more prominently at high temperatures and concentrations due to Ce^{3+} – Ce^{3+} interactions. The results agree with other results [20, 21]. The 5d state is the excited state with crystal field splitting that split the levels into 2 to 5 component (18000 cm^{-1}) and the lowest 4f level is the ground state that split into two component $^2F_{7/2}$ and $^2F_{5/2}$ due spin orbit coupling (2000 cm^{-1}). Ce^{3+} shows broad band shown in the energy level diagram Figure 6. Therefore emission of Ce^{3+} ions originates from the lowest crystal fields components of the 5d state to these terminating ground state levels. The strong intensity in excitation and emission spectra is observed when synthesized by WCS method rather than SSD and CS routes. Characteristic feature of these phosphors is 327 nm emission wavelength, which is in UV region and useful in phototherapy lamps.

There are two cation sites Na^{2+}, Mg^{2+} in $NaMgF_3$ in nearby vacancies. Taking into account the ionic radii of Ce^{3+} (1.15A^o), Na^{2+} (1.18A^o), and Mg^{2+} (0.86A^o) ions one could expect Ce^{3+} to substitute for Na^{2+}, charge compensation is required. The substitution of Ce^{3+} for Mg^{2+} may be less probable because of ion size mismatch. Electron spin resonance will confirm their assignment.

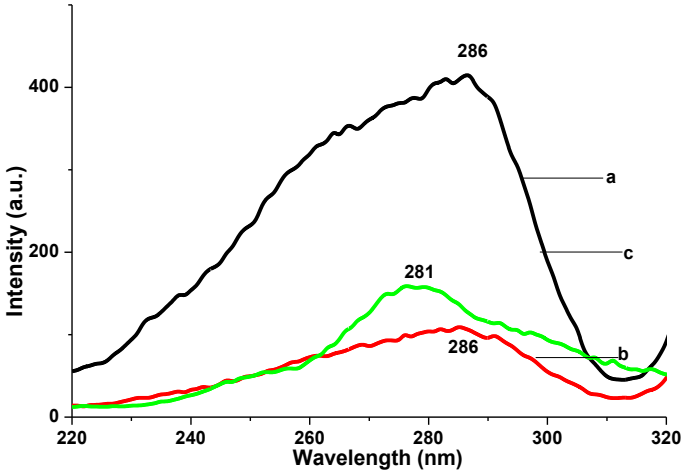

Figure 4: Excitation spectra of $NaMgF_3$:$Ce_{2m\%}$ at λ_{em}=327nm synthesized by a)WCS,b)SSD,C)CS

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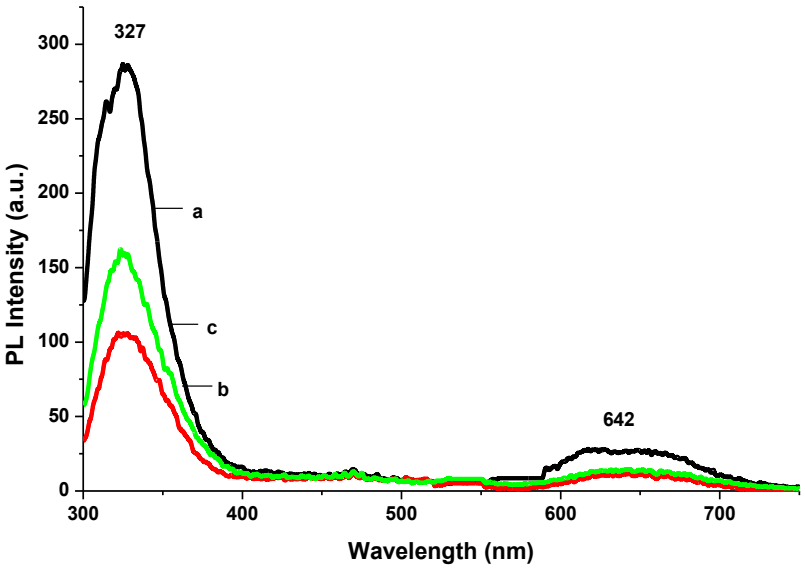

Figure 5: Emission spectra of $NaMgF_3$:Ce $_{2m\%}$ monitored at λ_{ex} = 286 nm synthesized by a) WCS, b) SSD, C) CS

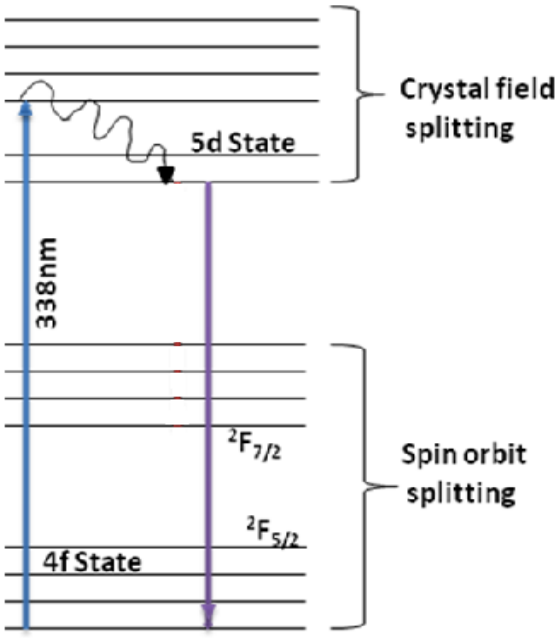

Figure 6: Schematic energy levels of Ce^{3+} in $NaMgF_3$

3.5 Luminescence in $NaMgF_3$: Eu^{3+}

Figure 7 shows excitation spectra of $NaMgF_3$:Eu^{3+}0.5 m% phosphor, the prominent peak is observed at around 395 nm with the small other peaks of f→ f transitions of Eu^{3+} at λ_{emi} = 594 nm, for the different routes of synthesis (WCS,SSD and CS route), showing change in intensities . The excitation bands appearing at longer wavelengths correspond to the characteristic f→ f transitions of Eu^{3+} with the main excitation line $^7F_0 \rightarrow {}^5L_6$ (395 nm). Figure 8 shows the emission spectra of $NaMgF_3$:Eu^{3+} phosphor synthesized by WCS and SSD and CS routes respectively, the prominent peak is observed at 594 nm due to $^5D_0 \rightarrow {}^7F_1$ magnetic dipole transitions and other emission at 616nm of Eu^{3+} is due to the hypersensitive $^5D_0 \rightarrow {}^7F_2$ electric dipole transition. The luminescence spectrum of Eu^{3+} ion is slightly influenced by surrounding legends of the host material because the transitions of Eu^{3+} involve only a redistribution of electrons within the inner 4f sub-shell. The transitions are found to be split into components, depending on the host matrix composition. The excitation of 395 nm is far away from Hg excitation which is a main characteristic of solid-state lighting, used in the lamp industry, which is exactly required by ultra-violet chip pumped multi-phosphor converted white LEDs. The strongest peak located at 594nm contributes the reddish-orange emission may be applicable LED lighting. Non radiative transitions may occur between $^5D_3$, $^5D_2$, $^5D_1$ to $^5D_0$, with probabilities from $^5D_3$ to $^5D_2$,

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from $^5D_2$ to $^5D_1$ and from $^5D_1$ to $^5D_0$. From level $^5D_0$ to level 7Fj (j = 0,1,2,3) radiative transitions is observed which is shown in energy level diagram Figure 9.

Despite availability of Mg^{2+}substitutional sites, there was no indication of Eu^{2+} emission. The PL emission spectra for divalent Eu are not observed in $NaMgF_3$ phosphor. Europium is incorporated in exclusively trivalent form when excitation wavelength was 395nm.The trivalent europium ion is very useful for studying the nature of metal coordination in various systems, owing to its non-degenerate emitting $^5D_0$ state. The results are supported by results seen in [22-24]. Belsare et. al. have observed the emission in $LiCaAlF_6$, $LiMgAlF_6$ $LiSrAlF_6$ $BaMgF_4$ host in Eu^{2+} line emission of Eu^{2+} had been reported for host $BaAlF_5$ [25-28]. Eu^{3+} emission in $NaMgF_3$, in this study, may be because high temperature treatment or any inert atmosphere is not used. It has been observed that there are no substantive differences between excitation and emission spectra of the all samples synthesized by WCS or SSD or CS route in the same atmospheric conditions. $NaMgF_3$:Eu^{3+} requires charge compensation because Eu^{3+} is believed to substitute on to the Na^{2+} site.

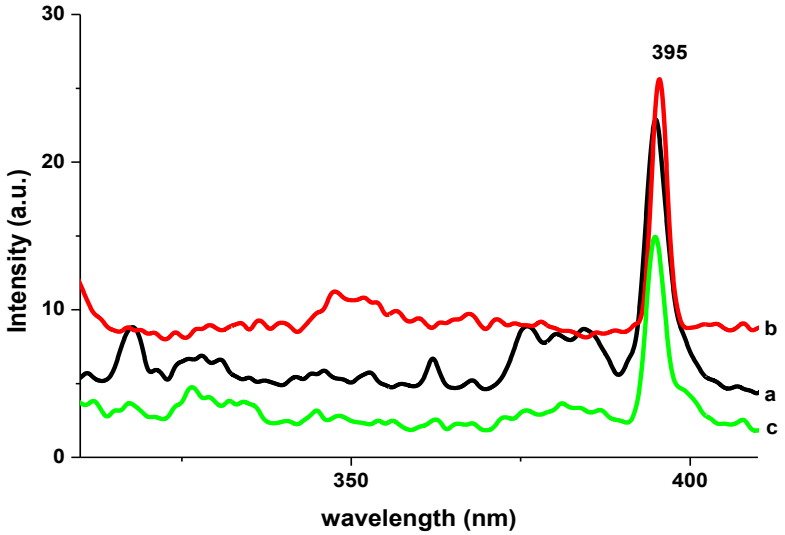

Figure 7: Excitation spectra of $NaMgF_3$: $Eu^{3+}_{0.5m\%}$ monitored at λ_{em}=594nm synthesized by a)WCS, b) SSD,C)CS

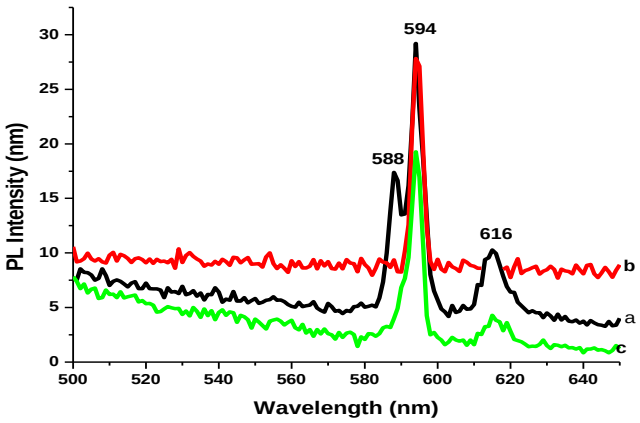

Figure 8: Emission spectra of $NaMgF_3$: $Eu^{3+}_{0.5m\%}$ monitored at λ_{ex} = 395nm synthesized by a) WCS, b) SSD, C) CS

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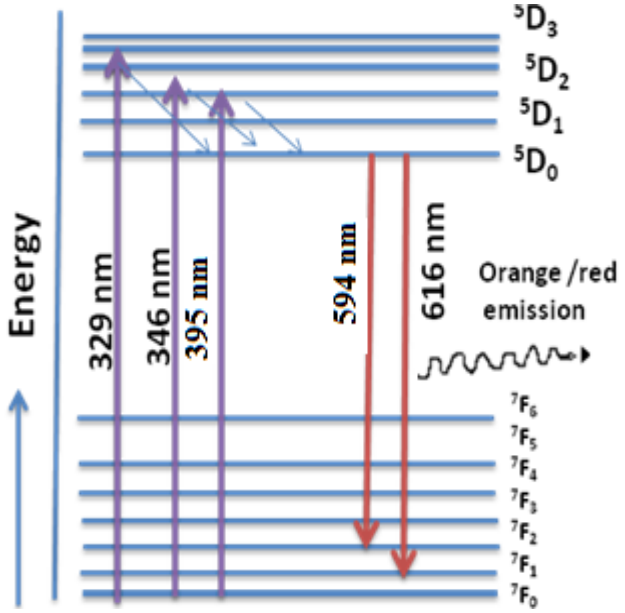

Figure 9: Schematic energy level diagram of Eu^{3+}in $NaMgF_3$

3.6 Comparison of phosphors when synthesized by Different routes

Table 1 shows the summary of PL in $NaMgF_3$ doped with Ce^{3+},Dy^{3+} and Eu^{3+} synthesized by different routes (WCS, SSD and CS).

Table 1: Summary of PL in $NaMgF_3$ pure and doped phosphors synthesized by different routes

Phosphor	Synthesis Route	λ_{exc} (nm)	$\lambda_{emission}$ (nm)	$I_{Max.}$ (a. u.)	Colour of Phosphor
$NaMgF_3$ Pure	WCS	358	388	68	UV
	SSD	358	389	23	UV
	CS	358	388	38	UV
$NaMgF_3$: Ce^{3+}	WCS	286	327,642	289 30	UV
	SSD	286	327,642	107 22	UV
	CS	286	327,642	162 22	UV
$NaMgF_3$: Eu^{3+}	WCS	395	594,616	30, 12	Orange-red
	SSD	395	594	29	Orange-red
	CS	395	594,616	20,05	Orange-red

4. Conclusion

Phosphors $NaMgF_3$ are successfully prepared by simple different routes without using any inert atmosphere. XRD patterns of $NaMgF_3$ prepared by WCS, SSD and CS methods are matching well with the standard ICSD 075291 and JCPDS file 15-0295, which is a direct evidence of the formation of the desired compounds. SEM of $NaMgF_3$ is showing the particles are in micro size 500 nm. The excitation bands of Ce^{3+} in $NaMgF_3$ are broad and remain at the same position, 286±5 nm when synthesized by WCS, SSD and CS route. All emission spectra are monitored at excitation wavelength of 286 nm showing emission at 327 nm (UV) and in red region at 642 nm. Excitation spectra of $NaMgF_3$:Eu^{3+} phosphor shows prominent peak at around 395±2 nm and in the $NaMgCl_3$: Eu^{3+} at around 397nm with the small other peaks of f→ f transitions of Eu^{3+}. Emission spectra are observed at 596±2 nm and other emission at 616±2 nm of Eu^{3+}. Photoluminescence studies by three routes (WCS, SSD and CS) showed there is no notable difference is seen in the PL spectra of $NaMgF_3$.

Captions of the figures

Figure 1: X-Ray diffraction pattern (XRD) of $NaMgF_3$ by WCS, SSD and CS route compared with standard XRD.
Figure 2: Excitation spectra of pure $NaMgF_3$ monitored at λ_{em}=389nm synthesized by a)WCS,b) SSD,C) CS
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