Temperature Dependent Optical Performance of the NaSr(PO$_3$)$_3$:Eu^{2+} Blue Phosphors

Chang yong Yoon, Sang ho Lee[*]

Department of Nuclear Medicine, Dongnam Institute of Radiological & Medical Sciences Cancer center

ABSTRACT

Eu^{2+} doped polyphosphate NaSr(PO$_3$)$_3$ blue-emitting phosphors were synthesized by the conventional solid state method in a reductive atmosphere. The phase formation of NaSr(PO$_3$)$_3$ phosphors were characterized by using the X-ray powder diffraction (XRD) measurement. The photoluminescence emission and excitation spectra of the NaSr(PO$_3$)$_3$:Eu^{2+} phosphor, and decay curves were measured. Under the near-UV excitation, the phosphor exhibits a band emission around 420 nm assigned to the 4f^{6}5d→f7(8S$^{7/2}$) transition of Eu^{2+}. The temperature dependent emission spectra and decay curves were measured to elevate the thermal properties of the Eu^{2+} doped phosphors. The as-prepared NaSr(PO$_3$)$_3$:Eu^{2+} phosphors show a strong temperature dependent performance, which can serve as a promising temperature sensor.

Keyword: NaSr(PO$_3$)$_3$, Eu^{2+}, Temperature dependent

I. INTRODUCTION

The divalent Eu^{2+} ion with 4f^7 electron configuration usually exhibits broad fluorescence emission due to f - d allowed transitions. The fluorescence of Eu^{2+} doped phosphors represent strong broad absorption bands in the ultraviolet (350 ~ 380 nm) or near - ultraviolet (380 ~ 420 nm) range and various emission wavelengths from ultraviolet to red[1-3]. The allowed 5d - 4f electrical dipole transition originates from the lowest band of the 4f^{6}5d^1 configuration to the 8S$^{7/2}$ grounded state of the 4f^7 configuration, resulting in a tun able broadband emission, which is highly dependent on the crystal field of the host lattices[4,5]. Thus, the luminescence properties of Eu^{2+} activated phosphors have been extensively investigated[6-8].

Polyphosphate, which are used as a representative series of host for rare earth activators, shows outstanding advantages such as environmental safety, high luminous efficiency and relatively low cost[9,10]. Therefore, these polyphosphate are suitable for the application of illumination and used as potential candidate as phosphor for light emitting diodes (LED) industry by structure engineering[11,12]. Particularly, the PO$_4$$^{3-}$ tetrahedral in the polyphosphate host with rigid structure is benefit for the formation and stabilization of the divalent Eu^{2+} [13,14] In addition, alkaline earth metal ions such as Sr^{2+}, Ba^{2+} are ideal ions to be suitable for Eu^{2+} substitution due to the similarity of ionic radii[15-19]. However, the luminescence efficiency of the Eu^{2+} doped polyphosphate is strongly influenced by the temperature and the bad thermal stability limits the application in white LED. Therefore, it is necessary to study the thermal stability of the Eu^{2+} doped polyphosphate and investigate the application of these kinds of phosphors in other field[20-22].

* Corresponding Author: Sang ho Lee   E-mail: riroom@hanmail.net   Tel: ***- ****- ****

Address: Department of Nuclear Medicine, Dongnam Institute of Radiological & Medical Sciences Cancer center

In the present work, Eu^{2+} doped $NaSr(PO_3)_3$ phosphors were prepared by conventional high-temperature solid state method. The phase and crystal structure are investigated in detail. Furthermore, the temperature- dependent luminescence spectra and decay curves measurement were measured and the activation energy was confirmed. Results show that $NaSr(PO_3)_3$:Eu^{2+} phosphor is a potential temperature sensor.

II. MATERIAL AND METHOD

1. Materials Synthesis.

The compounds $NaSr(1-x)(PO_3)_3$:xEu^{2+} (x = 0.1% ~ 5%) were synthesized by the conventional solid-state reaction. Stoichiometric amounts of Na_2CO_3, $SrCO_3$, $NH_4H_2PO_4$ and Eu_2O_3 were mixed and completely ground in an agate mortar with a small quantity of ethanol. Firstly, the mixture was heated to 400 ^oC and kept at this temperature for 5 h. After regrinding, they were put into crucibles and heated at 650 ^oC for 10 h. The as-prepared Eu^{3+} doped materials were grounded heated at 650 ^oC with a reduction atmosphere for 24 h. Finally, the reduced Eu^{2+} doped samples were obtained after cooling down the furnace to room temperature naturally.

2. Materials Characterization.

A X-ray Rigaku D/Max diffractometer operating at 40 kV, 30 mA and equipped by Cu Kα radiation (λ= 1.5405 Å) was used to record the X-ray diffraction (XRD) data. A 450 W Xe lamp dispersed by 25 cm monochromator (Acton Research Corp. Pro-250) was used as a light source for excitation and emission spectra. The luminescence was dispersed by a 75 cm monochromator (Acton Research Corp. Pro-750) and observed with a photomultiplier tube (PMT) (Hamamatsu R928). The sample was cooled in a closed cycle helium cryostat and measurements were taken in the temperature range of 10 ~ 300 K. The luminescence decay curves were excited by the 355

nm-pulsed Nd-YAG laser (Spectron Laser Sys. SL802G)and collected by means of a 500 MHz Tektronix DPO 3054 oscilloscope.

III. RESULT and DISCUSSION

1. Phase identification

Fig. 1(a) depicts the XRD patterns of the as-prepared $NaSr(1-x)(PO_3)_3$:xEu^{2+} (x = 0.1% ~ 5%) phosphors along with the standard PDF card of $NaSr(PO_3)_3$ (PDF# 25-0857). All the reflection peaks are indexed well with the standard $NaSr(PO_3)_3$ phase with a triclinic space group of P-1(2). No detectable diffraction peaks corresponding to impurity phases are observed. As shown in Fig. 1(b), the XRD peaks located at 25.2^o, corresponding to the (200) plane, show slightly shift to the higher angle direction with the Eu^{2+} content increasing, indicating that the Eu^{2+} with smaller ion radio (r = 1.25 Å, CN = 8) completely substitutes the Sr^{2+} ions (r = 1.26 Å, CN = 8) in the lattice of the as-prepared phosphors.[23,24]
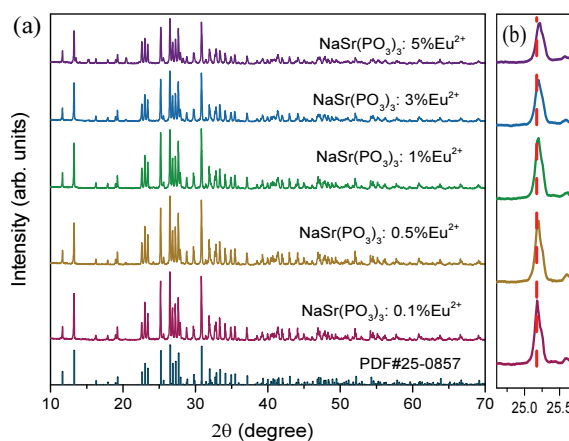

Fig. 1. (a) The XRD patterns of $NaSr_{(1-x)}(PO_3)_3$:xEu^{2+} (x = 0.1% ~ 5%) as a function of Eu^{2+} contents and compared with the standard XRD data of $NaSr(PO_3)_3$ (PDF#No.25-0857).(b) Magnified XRD patterns in the region of 25 ~ 25.5 degree for $NaSr_{(1-x)}(PO_3)_3$:xEu^{2+} as a function of Eu^{2+} contents[25].

Fig. 2 shows the crystal structure of $NaSr(PO_3)_3$ in the super cell matrix with double cell X. the structure contains several $(PO_3)nn^-$ chains with a period of tetrahedra PO_4 by corner shared, and forming long belts running zig-zags perpendicular to axis c, the Na^+ and Sr^{2+} ions occupy the structural tunnels between the chains in the three-dimensional crystal network, coordinating to six and eight terminal oxygens, respectively.
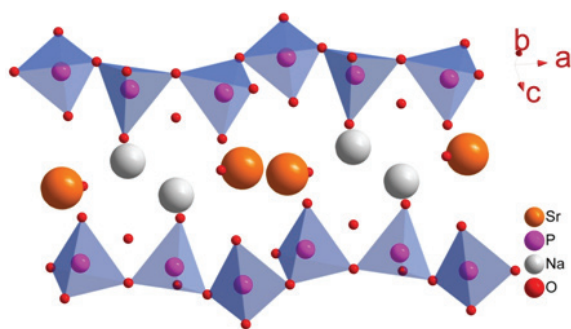

Fig. 2. Crystal structure of $NaSr(PO_3)_3$

2. Optical properties of $NaSr(PO_3)3:x\%Eu^{2+}$ phosphors

Fig. 3 shows the typical excitation and emission spectra of $NaSr(PO_3)_3:3\%Eu^{2+}$ phosphor at 10 K. The excitation spectrum monitored at 421 nm consists of a structureless broad band with several peak features in the wavelength region 200 ~ 400 nm with a maximum at 325 nm, which consisted of well resolved bands of the $4f^65d^1$ multiplets of the Eu^{2+} excited states at low temperature. Therefore, the excitation band could be assigned to the d - f transition of Eu^{2+}. Obviously, the excitation spectrum covers UV to blue region indicating that $NaSr(PO_3)_3:3\%Eu^{2+}$ phosphors can be used as a near UV chips excited blue-emitting phosphor. The emission spectrum under the excitation of 320 nm consist of a broad asymmetric band peaked at 425 nm in the range of 400 ~ 480 nm, which corresponds to the Eu^{2+} allowed $4f^65d^1$ - $4f^7$ electronic transitions.
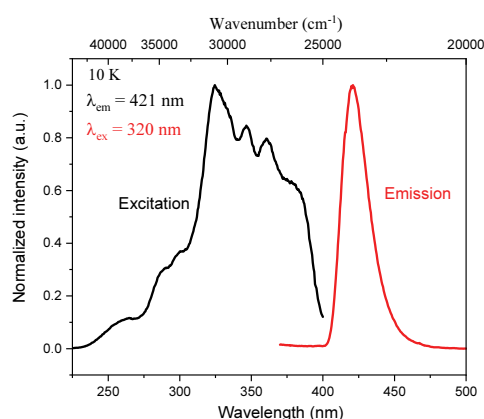

Fig. 3. The normalized PL (λ_{ex} = 320 nm) and PLE (λ_{em} = 421 nm) spectra of $NaSr(PO_3)_3:3\%Eu^{2+}$ at 10 K.

Fig. 4(a) shows the PL intensity of $NaSr(PO_3)_3:Eu^{2+}$ as a function of doped Eu^{2+} concentration. As shown in Fig. 4(b), the luminescence intensity increases with increasing Eu^{2+} concentration and reaches a maximum intensity at 3 mol%. However, when the doping concentration is higher than 3 mol%, the luminescence intensity dramatically decreases due to the concentration quenching, mainly caused by the nonradiative energy transition among the identical activator Eu^{2+} ions[18]. Generally, nonradiative transition was attributed to radiation reabsorption, exchange interaction or electric multipolar interaction[26]. Blasse pointed out that the critical distance (R_c) between the nearby Eu^{2+} ions can be estimated by the following Eq. (1):

$$R_c = 2\left(\frac{3V}{4\pi x_c N}\right)^{1/3} \qquad (1)$$

Where x_c is the critical concentration, V is the volume of unit cell and Z represents the number of formula units per unit cell. Here, N=1.5, x_c=0.03, and V=365.19 Å^3. Thus, the critical transfer distance R_c is calculated to be 24.93 Å. Generally, the exchange interaction commonly occurs in the forbidden transition, that is to say, the nonradiative transition among the Eu^{2+} ions in $NaSr(PO_3)_3:3\%Eu^{2+}$

phosphors should be controlled by electric multipolar - multipolar interaction. The interaction type between the Eu^{2+} sensitizers can be calculated by the following Eq. (2):

$$\frac{1}{x} = \frac{k}{1 + \beta(x)^{\theta/3}} \qquad (2)$$

where I represents the emission intensity, x represents the activator concentration, k and β are constants for each type of interaction under the same measurement conditions. The value of θ is a function of multipole - multipole interaction, where θ = 6, 8, and 10 represent electric dipole - dipole, dipole - quadrupole, and quadrupole - quadrupole interactions, respectively and θ = 3 stands for the energy transfer between nearest Eu^{2+} ions[27]. The relationships of logI/x vs logx are shown in the inset of Fig. 4(c) and the slope of the fitting line is -0.8833. Hence, the value of θ is calculated to be to 2.41 and approximately equal to 3, which means that the dominant concentration quenching mechanisms between Eu^{2+} ions occur via energy transfer between nearest Eu^{2+} ions[25,28].

In order to investigate the thermal stability of NaSr(PO$_3$)$_3$:3%Eu^{2+}, PL spectra NaSr(PO$_3$)$_3$:3%Eu^{2+} as a function of temperature are measured under the excitation of the 320 nm Xe lamp. As shown in Fig. 5(a), the integrated PL intensity maintains a stable magnitudein the range of 10 ~ 90 K as shown in Fig. 5(b). With the temperature further elevated, the PL intensity decreases sharply due to thermal quenching. The quenching temperature T50 is usually defined as the temperature at which the intensity is half of the maximum intensity.

In this case, the T50 of NaSr(PO$_3$)$_3$:3%Eu^{2+} is evaluated at 165 K. In trivalent rare 4f^7 system, the electrons in 4f orbital is shield and barely influenced by the local environment, and the dominant nonradiative relaxation path is the multiphonon relaxation.
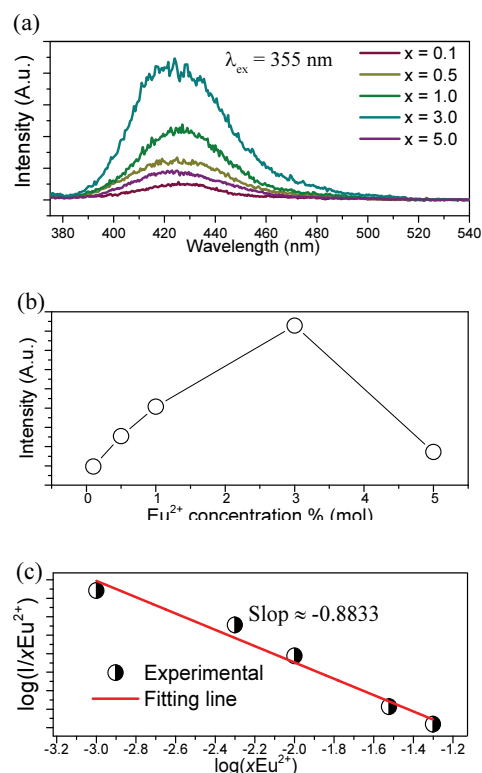
Fig. 4. (a) PL spectra of NaSr$_{(1-x)}$(PO$_3$)$_3$:xEu^{2+} (x = 0.1% ~ 5%) under the excitation of 355 nm; (b) The corresponding PL intensities as a function of Eu^{2+} contents. (c) Concentration dependence of lg(I/xEu^{2+}) on lg(xEu^{2+})for NaSr$_{(1-x)}$(PO$_3$)$_3$:xEu^{2+} (x = 0.1% ~ 5%) phosphor[25].

On the other hand, the 5d electrons are unshielded in the divalent Eu^{2+} of 4f^{5}d electronic shell and the optical properties of Eu^{2+} are very sensitive to the environment, in particular influenced by the temperature.

The thermal quenching mechanism can be explained by the configuration coordinate diagram as shown in Fig. 6. The red parabola and the black parabola represent the excited state and ground state, respectively. The parabola of the excited state deviates from the equilibrium position and have a crossover point C with the parabola of the ground state. Thus, the electron in the excited state can reach the ground state nonradiatively by the red vertical broken arrows as shown in Fig. 6.
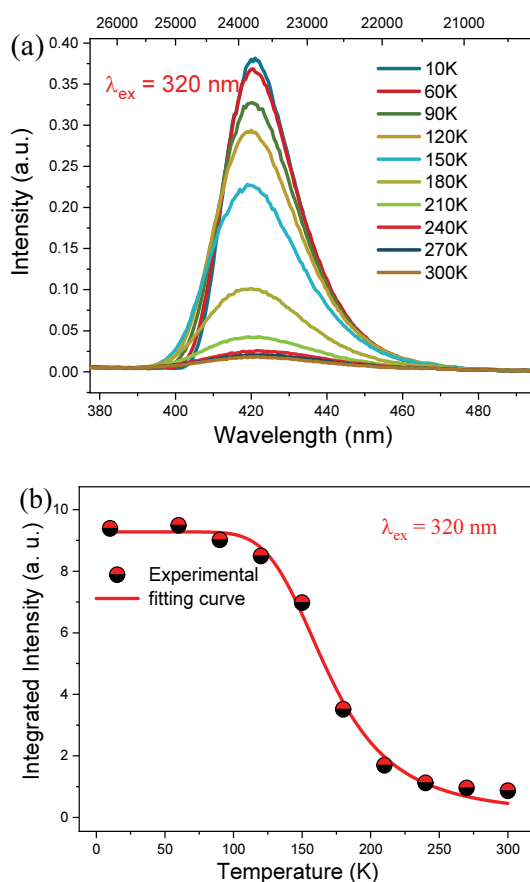
Fig. 5. (a) Temperature-dependent PL spectra of NaSr(PO₃)₃:3%Eu²⁺ under 320 nm excitation. (b) Integrated emission intensity as a function of temperature in the range of 10 ~ 300 K, the red curve represents the fitting result according to the Arrhenius-type Mott equation.
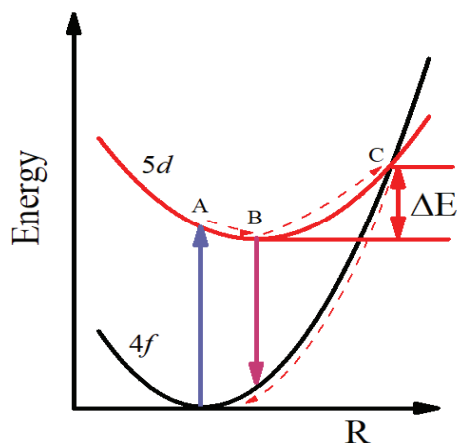

Fig. 6. The schematic configuration coordinate diagram for the excited state 5*d* and ground state 4*f*. The red dash curve shows the nonradiative relaxation route.

The energy difference between lowest excited state B and the cross point C is name as activation energy △, and it can be obtained from the following Eq. (3):

$$I_T = \frac{I_0}{1 + C_1 \exp\left(-\dfrac{\Delta E}{kT}\right)} \tag{3}$$

Similarly, the temperature dependent lifetime also have the following Eq. (4):

$$\tau_T = \frac{\tau_0}{1 + C_2 \exp\left(-\dfrac{\Delta E}{kT}\right)} \tag{4}$$

As shown in Fig. 7(a), the decay times cannot be fitted by single-exponential function and as shown in Fig. 7(b) Calculated average lifetimes of NaSr(PO₃)₃:3%Eu²⁺ as a function of temperature in the range of 10 ~ 300K, therefore, the average decay time can be employed to calculate the lifetime by the following Eq. (5):

$$\tau = \frac{\int_0^\infty t I(t)\,dt}{\int_0^\infty I(t)\,dt} \tag{5}$$

According to the above equations, the activation energies are calculated to be 806 and 763 cm⁻¹ for NaSr(PO³)³:3%Eu²⁺ calculated through temperature-dependent luminescence intensities and decay times, respectively. Compared with other published oxides blue phosphors such as $Ca_3SiO_4Cl_2$:Eu²⁺ (1281 cm⁻¹)[6], $Ba_3BP_3O1_2$:0.01Eu²⁺ (3465 cm⁻¹)[5], $Sr_9Mg_{1.5}(PO_4)_7$:0.06Eu²⁺ (2417 cm⁻¹)[20], the obtained borate blue phosphors NaSr(PO₃)₃:3%Eu²⁺ depicts sensitive temperature dependent characters. Due to the decay measurement is barely influenced by the instrumental conditions, it is suggests that Eu²⁺-doped NaSr(PO₃)₃ is an acceptable temperature senor for thermometry by calculating the temperature dependent lifetime.
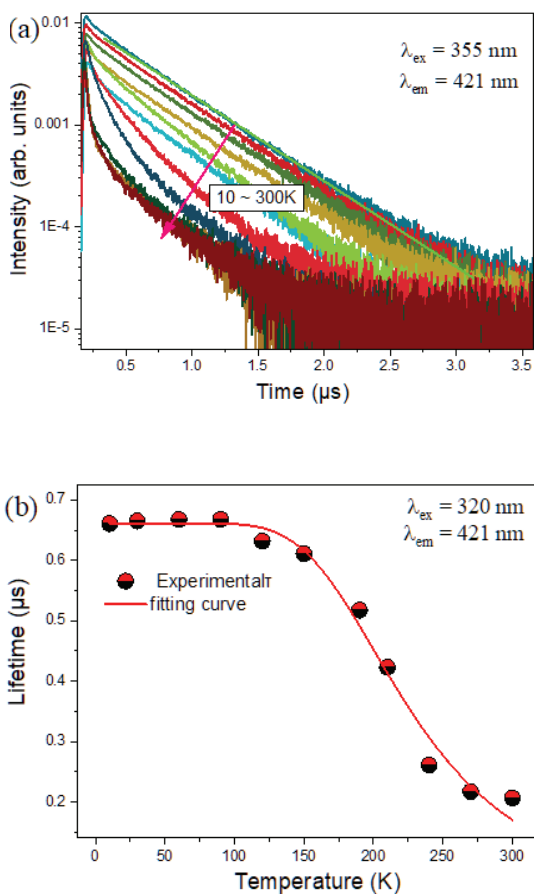
Fig. 7. (a) Decay curves of NaSr(PO₃)₃:3%Eu²⁺ as a function of temperature. (b) Calculated average lifetimes of NaSr(PO₃)₃:3%Eu²⁺ as a function of temperature.

IV. CONCLUSION

Polyphosphate NaSr(PO₃)₃:Eu²⁺ doped samples were prepared via conventional high-temperaure solid state reaction. The blue-emitting NaSr(PO₃)₃:Eu²⁺ phosphors can be efficiently excited by the UV to blue light and show a narrow emission band in the range of 400 ~ 480 nm at 10 K, assigned to the allowed Eu²⁺ d - f transition. The thermal quenching properties of NaSr(PO₃)₃:Eu²⁺ phosphors were evaluated according to the temperature dependent emission spectra and decay curves. The fitting results illustrate that NaSr(PO₃)₃:Eu²⁺ phosphors depict very low thermal stability ($\triangle$ 800 cm⁻¹), when compared with other reported oxides blue phosphors. The sensitive temperature dependent features demonstrate that NaSr(PO₃)₃:Eu²⁺ phosphors have potential to serve as temperature sensor candidates.

Acknowledgement

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NaSr(PO$_3$)$_3$:Eu^{2+} 청색 형광체의 온도 의존적 형광 특성

윤창용, 이상호*

동남권원자력의학원 핵의학과

요 약

고상법을 이용하여 Eu 이온이 첨가된 다중인산염 NaSr(PO$_3$)$_3$형광체를 합성하고 탄소열환원법을 통하여 NaSr(PO$_3$)$_3$:Eu^{2+} 형광체를 완성하였다. X선 회절 측정(XRD)을 통하여 형광체의 결정상을 확인하였다. NaSr (PO$_3$)$_3$:Eu^{2+} 형광체의 Eu^{2+} 농도 변화에 따른 발광 방출 및 여기 스펙트럼과 수명시간을 측정하였다. 320 nm 근자외선 여기에서 NaSr(PO$_3$)$_3$:Eu^{2+} 형광체는 Eu^{2+}의 4f^{6}5d → f^7(8S$^{7/2}$) 전이에 따른 420 nm 중심의 밴드를 방출하였다. Eu^{2+} 첨가 된 형광체의 열적 특성을 증가시키기 위해 온도 의존적 방출 스펙트럼과 형광 감쇠 곡선을 측정하고 형광체의 온도 의존성을 설명하였다. 본 연구로 희토류 이온이 첨가된 NaSr(PO$_3$)$_3$:Eu^{2+}는 디스플레이, 검출기 등에 온도 센서 역할을 할 수 있는 우수한 온도 의존성을 보여준다.

중심단어: NaSr(PO$_3$)$_3$, Eu^{2+}, 온도 의존성

연구자 정보 이력

	성명	소속	직위
(제1저자)	윤창용	동남권원자력의학원 핵의학과	방사선사
(교신저자)	이상호	동남권원자력의학원 핵의학과	방사선사