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Photoluminescence investigation of novel $KCaPO_4:Sm^{3+}$ phosphors for n-UV based solid state lighting Prepared by wet chemical synthesis

C. M. Nandanwar[1] · N. S. Kokode[2] · A. V. Nande[3] · C. D. Mungmode[4] · A. N. Yerpude[1] · R. M. Yerojwar[5] · S. J. Dhoble[6]

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Abstract
The wet chemical method was used for the first time to synthesize novel $KCaPO_4:Sm^{3+}$ phosphor. The characteristics of XRD, morphology and photoluminescence were thoroughly investigated. The hexagonal structure of $KCaPO_4$ is confirmed by the XRD analysis. The $KCaPO_4:Sm^{3+}$ phosphor emission are peaks located at 565 nm, 599 nm and 646 nm under excited at 403 nm. Concentration quenching was shown to occur at 1 mol% of Sm^{3+} ions. It was found that concentration quenching occurred as a result of quadrupole–quadrupole interaction according to Dexter's theory. The CIE Chromaticity coordinate of the prepared phosphor was located in the orange region around (0.602, 0.395) with high color purity. The current study suggests that $KCaPO_4:Sm^{3+}$ phosphors could potentially represent a promising n-UV converter material for solid-state lighting applications.

Keywords Photoluminescence · XRD · Wet chemical method · Phosphor · Solid state lighting

1 Introduction

The optical material has more attention from researchers and the scientific community for developing materials for w-LED (Yam and Hassan 2005; Yerpude et al. 2019a; Nandanwar et al. 2022; Ramteke et al. 2021). The w-LEDs are considered the next generation of solid

✉ C. M. Nandanwar
chandrahasyanandanwar@gmail.com

[1] Department of Physics, N. H. College, Bramhapuri, Chandrapur 441206, India

[2] N. H. College, Bramhapuri, Chandrapur 441206, India

[3] Guru Nanak College of Science, Ballarpur 442701, India

[4] Department of Physics, M. G. Arts, Science & Late N. P. Commerce College, Armori 441208, India

[5] Department of Physics, Mohasinbhai Zaweri Mahavidyalaya, Desaiganj (Wadsa) 441207, India

[6] Department of Physics, RTM Nagpur University, Nagpur 440033, India

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state lighting devices, and they are on their way to replacing traditional incandescent and fluorescent lamps due to benefits such as environmental eco-friendly and energy savings (Li et al. 2015; Yerpude et al. 2019b). Due to their no pollution, reliability, long lifetime, high brightness, quick response and low production cost, phosphor based white light emitting diodes have a wide range of applications. The conversion of phosphor to w-LED has a huge advantage and great economic benefits, attracting people all over the world. As a response, it was essential to achieve high quality and colour rendering index of solid state lighting to show higher thermal stability.

Researchers worked hard to develop an efficient and stable phosphor material that could be activated by n-UV and emit visible light. Hence, after examining the literature on phosphates, we are drawn to the phosphate family of materials due to their easy-to-synthesis process (Nandanwar et al. 2023a; Hou et al. 2016), high thermal stability and chemical stability (Nandanwar et al. 2023b; Tao et al. 2014). Researchers investigated such as $SrLa_2Al_2O_7:Eu^{3+}$ (Devi et al. 2022), $Ba_3GdP_3O_{12}:Dy^{3+}$ (Chhillar et al. 2022a), $SrGdAlO_4:Tb^{3+}$ (Chhillar et al. 2022b), $BaYZn_3AlO_7:Er^{3+}$ (Hooda et al. 2022), $BYO:Er^{3+}$ (Hooda et al. 2021a) and $Ba_3Y_4O_9$: Dy^{3+} (Hooda et al. 2021b) which had good optical characteristics and were proposed as phosphors materials for use in w-LEDs. The use of Sm^{3+} ion dopant in host phosphors for solid-state lighting has several significant advantages. Solid-state lighting offers high color rendering, narrow emission lines, red emission for warm white light, energy efficiency, stability, and compatibility with LED technology. These properties make Sm^{3+} ions doped phosphors an attractive choice for developing advanced solid-state lighting solutions with improved performance and reduced environmental impact (Yerojwar et al. 2022a; Wang et al. 2012).

Rare earth ions have played an important role in the development of several commercial phosphors and are presently being studied for their potential to improve the optical properties of materials (Nandanwar et al. 2023c; Yerpude and Dhoble 2013). C. M. Nandanwar et al. (Nandanwar et al. 2023d) reported a wet chemical technique for producing $Ba_3(PO_4)_2:RE=Dy^{3+}$, $Sm^{3+}$and Eu^{3+} phosphors. P. Chhillar et al. (Chhillar et al. 2022c) $SrGd_2Al_2O_7:Er^{3+}$ phosphor has been thoroughly studied due to its relevance in traditional solid state lighting. The wet chemical method is easy to prepare and has low sintering temperature, low-cost methods, eco-friendly and low energy (Nandanwar et al. 2023e). In this study, the $KCaPO_4:Sm^{3+}$ phosphors were first time prepared by a wet chemical technique. We carefully investigated the XRD, photoluminescence properties, morphology and CIE coordinates. The $KCaPO_4:Sm^{3+}$ phosphors were synthesized by a wet chemical technique, and the prepared phosphors material has great potential in solid-state lighting.

2 Experimental method

The $KCaPO_4:Sm^{3+}$ phosphors were successfully synthesized by the wet chemical method. The initial raw materials included potassium nitrate (loba, 99%), calcium nitrate (loba, 99%), ammonium dihydrogen phosphate (loba, 99%) and samarium oxide (loba, 99.9%). AR-grade materials and chemicals are used throughout. To stoichiometrically measure the samples first, the chemical was weighed in a beaker. Sm_2O_3 dopants were then weighed and placed in a test tube and the mixture (HNO_3) was heated and converted into $Sm(NO_3)_3$. All samples and dopants were mixed in a beaker and water was added. It was then stirred for 30 min until all the liquid became transparent. All the samples were mixed and then autoclaved in a hot air oven at 100 °C for 10 h. This crystalline powder was ground into

a fine powder with a pestle and mortar. Finally, the powder was annealed in a 600 °C furnace for three hours. After annealing, the powders were slowly cooled to room temperature naturally in the furnace and were ground into powders in the agate mortar for further characterization. The PL excitation and emission spectra, all photoluminescence properties detected by Shimadzu RF5301 PC Spectroflurophotometer. All the measurements were carried out at room temperature. In the Investigation, the phase purity is determined by using an X-ray diffraction pattern in an advanced Rigaku miniflex X-Ray diffractometer.

3 Results and discussion

3.1 X-ray Diffraction

The analysis of structural parameters, Phase verification and Crystalline structure of synthesized $KCaPO_4$ phosphor was studied by powder X-ray diffraction using with range diffractometer with 2θ in of 20°–90°, prepared by Wet chemical method. Figure 1 (a) represents the JCPDS No. 00-033-1002 (b) $KCaPO_4$ host (c) $KCaPO_4$ 0.3% Sm^{3+} phosphor. The measured diffraction peak of the phosphor was a good agreement with the JCPDS No. 00-033-1002 with a hexagonal phase and space group P-3m1. The XRD pattern demonstrates that all diffracted peaks of prepared phosphor are very sharp and intense, which says that the synthesized sample was crystalline and the form was homogeneous (Ye et al. 2010; Guo et al. 2009). The obtained pattern of $KCaPO_4$ phosphor has no additional other phases and no other impurity phases in the composition.

3.2 SEM study of $KCaPO_4$ phosphor

The morphology of wet chemical synthesis was studied using SEM images at various resolutions. Figure 2a and b displays an SEM picture of $KCaPO_4$ phosphor at various

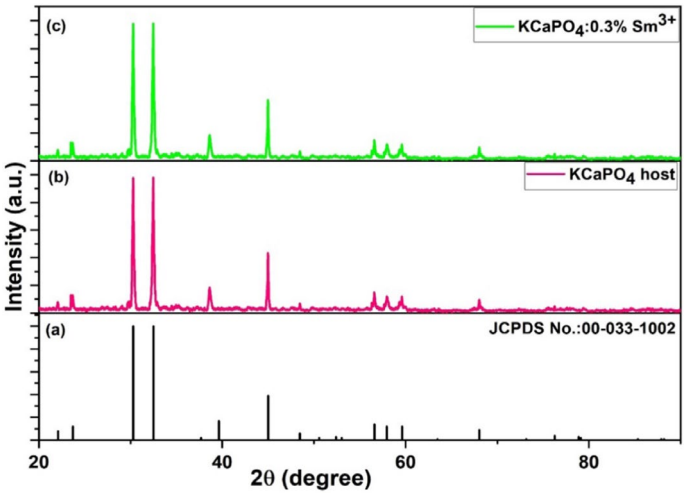

Fig. 1 **a** XRD pattern of JCPDS No. 00-033-1002, **b** $KCaPO_4$ host, **c** $KCaPO_4:Sm^{3+}$ phosphor

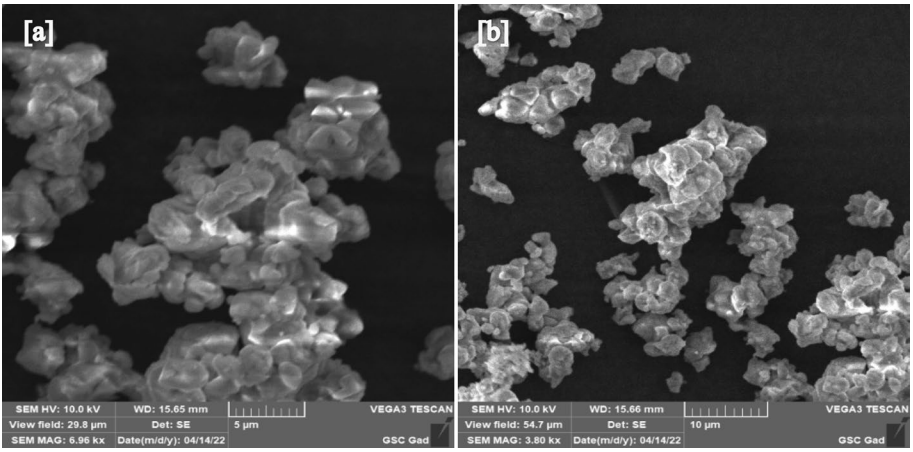

Fig. 2 **a** and **b** SEM images of $KCaPO_4$ phosphor at different magnification

magnifications. The phosphor microstructure is comprised of irregular grains with agglomeration in the area under the image. The formation of agglomerates can lead to variations in particle size and local environment, resulting in a broader emission spectrum and a shift in the peak wavelength. The average size of the as synthesized phosphor was near about 5–10 µm. As a result, they may be utilized for a wide range of lighting applications (Nandanwar et al. 2023f).

3.3 Photoluminescence properties of $KCaPO_4:Sm^{3+}$ phosphor

The Photoluminescence properties of $KCaPO_4:Sm^{3+}$ phosphor were investigated for solid state lighting applications. The excitation spectra of synthesized phosphor in the wavelength range 340 nm to 430 nm. The excitation spectra of the $KCaPO_4:Sm^{3+}$ phosphor are shown in Fig. 3, under the emission spectra at 599 nm. The excitation Peaks located around 345 nm, 362 nm, 376 nm, 403 nm and 418 nm are ascribed to Sm^{3+} ion transition from

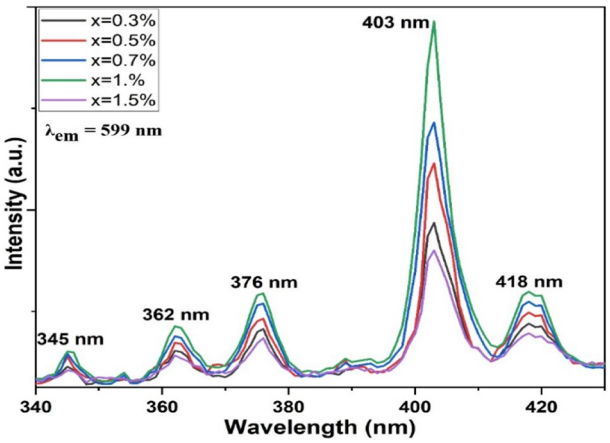

Fig. 3 Excitation spectra of $KCaPO_4:Sm^{3+}$ phosphor ($\lambda_{em} = 599$ nm)

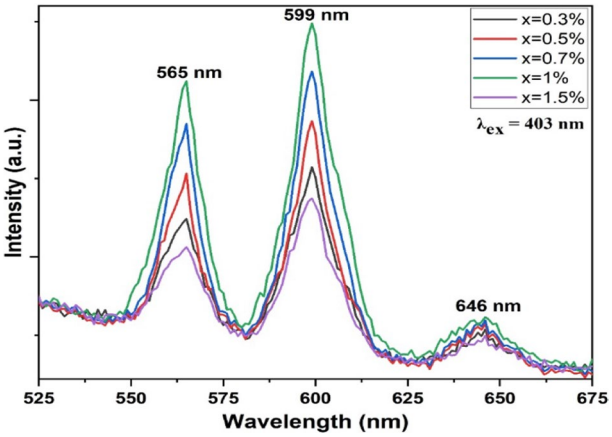

Fig. 4 Emission spectra of $KCaPO_4:Sm^{3+}$ phosphor excitation peak at 403 nm

Table 1 Estimated Stokes shift observed for all transitions $KCaPO_4:Sm^{3+}$ phosphor

Excitation (nm)	Emission (nm)	Stokes shift (cm^{-1})
345	565	11,286
362	599	12,291
376	646	13,506
403		9925
418		10,930
		12,144
		8897
		9901
		11,116
		7115
		8119
		9334
		6224
		7229
		8444

ground state $^6H_{5/2}$, manifolds of to $^4H_{9/2}$, $^4D_{3/2}$, $^4D_{1/2}$, $^4F_{7/2}$ and $^6F_{5/2}$ excited state transition (Xu et al. 2014; Yerojwar et al. 2023a; Sheoran et al. 2021).

Figure 4 Show the emission spectra of $KCaPO_4:Sm^{3+}$ phosphors. The three significant emission peaks are located in the wavelength region between 525 to 675 nm. Under the excitation spectra at 403 nm, the emission spectrum consists of three characteristic peaks around 565 nm 599 nm and 646 nm, indicating yellow, orange-red and strong red emission colours respectively. This band is attributed due to Sm^{3+} ions $^4G_{5/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$ and $^4G_{5/2} \rightarrow {}^6H_{9/2}$ transition (Nandanwar and Kokode 2022; Duan et al. 2020; Yerojwar et al. 2022b). The 599 nm wavelength has the maximum emission intensity among these other bands

The estimated Stokes shift for all observed excited and emission transitions are shown in Table 1. The highest Stokes shift is observed for $^6H_{5/2} \rightarrow {}^4H_{9/2}$ excitation transition and

$^4G_{5/2} \rightarrow {}^6H_{9/2}$ emission transition which is 13,506 cm^{-1} (corresponding to 1.68 eV). Also, the Stokes shift between prominent (highest intensity) excitation transition $^6H_{5/2} \rightarrow {}^4F_{7/2}$ and prominent (highest intensity) emission transition $^6H_{5/2} \rightarrow {}^4F_{7/2}$ measured for the same excitation wavelength is 8119 cm^{-1} or 1.01 eV.

We observed the effect of concentration doping Sm^{3+} ions on the emission intensity of $KCaPO_4:Sm^{3+}$ phosphor as represented in Fig. 5. The photoluminescence emission intensity increases with the concentration of Sm^{3+} ions from 0.3 mol % up to 1 mol % and subsequently decreases with the concentration of Sm^{3+} ions. This decreases intensity because of concentration quenching of Sm^{3+} ions. The maximum intensity was observed at 1 mol %. The concentration quenching was because the interaction between the Sm^{3+} ions continues to increase & it leads to emission intensity decreases (Xiang et al. 2019; Nandanwar et al. 2023g; Zhang et al. 2008).

Figure 5 clearly depicts that the peaks centered at 565 nm ($^4G_{5/2} \rightarrow {}^6H_{5/2}$) and 599 nm ($^4G_{5/2} \rightarrow {}^6H_{7/2}$) have prominent concentration quenching after 1% molar concentration of Sm^{3+} ions in $KCaPO_4$ host matrix. As the obvious reason for the concentration quenching is the multipole-multipole energy transfer or interaction between Sm^{3+} ions, one can estimate the multipolar energy transfer using equation (Uitert 1967).

$$\frac{I}{x} = \frac{K}{1 + \beta(x)^{\theta/3}}$$

where I is the highest emission intensity at given concentration x. K and β are the constant for a given host which is considered to be constant at the same excitation conditions. The estimated values of θ decides the corresponding interaction responsible for the concentration quenching – the possible values of θ are 6, 8, and 10 corresponding to dipole–dipole, dipole-quadrupole and quadrupole–quadrupole interactions, respectively (Yerojwar et al. 2023b). For simplicity, the above formula can reduce by considering ln on both side and plotting as ln(x) vs ln(I/x) which gives a straight line in the quenched region as shown in Fig. 6. The estimated slope value of the straight line is analogous to (-θ/3). For the given sample, the estimated value of θ/3 for our sample 2.689 for $^4G_{5/2} \rightarrow {}^6H_{5/2}$ transition and 2.465 for $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition. Thus, the estimated values of θ are 8.067 and 7.395 for $^4G_{5/2} \rightarrow {}^6H_{5/2}$ transition and $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition respectively. These values are closer to 8 for both the transitions suggesting concentration quenching mechanism of $KCaPO_4:Sm^{3+}$ phosphor is due to quadrupole–quadrupole interactions.

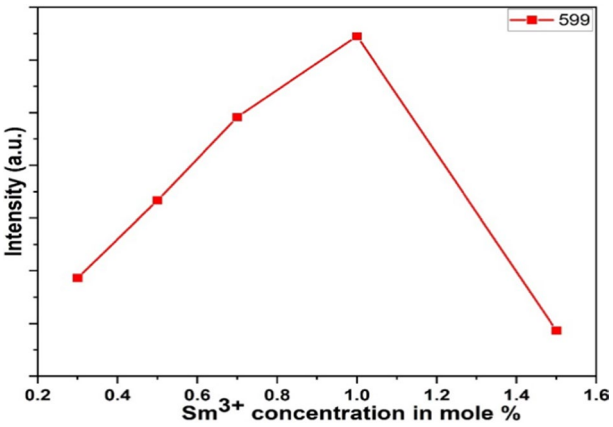

Fig. 5 Emission intensity at 599 nm as a measure of Sm^{3+} ion concentration in $KCaPO_4:Sm^{3+}$ phosphor

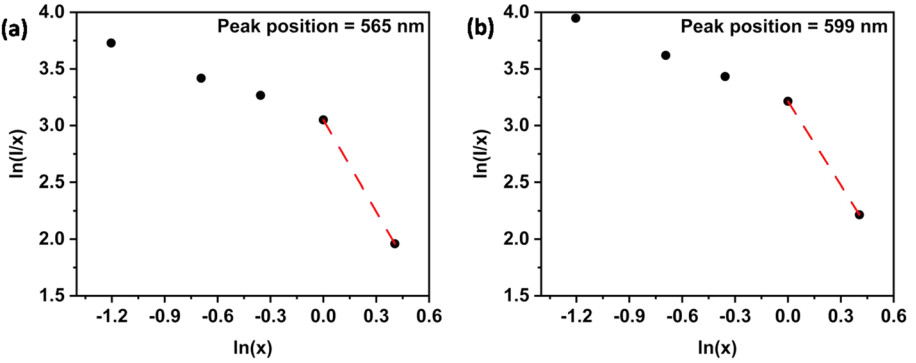

Fig. 6 Analysis curve for multipolar energy transfer curve for $KCaPO_4:Sm^{3+}$ phosphor. **a** The curve of ln(x) vs ln(I/x) for $^4G_{5/2} \rightarrow {}^6H_{5/2}$ transition **b** The curve of ln(x) vs ln(I/x) for $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition

The energy level diagram of Sm^{3+} ions in Fig. 7 shows the excitation and emission process. To begin, a non-radiative relaxation process takes place during the transition from $^4H_{9/2}$ and $^6G_{5/2}$ levels. After that, yellow and orange-red emission appear, attributed to intraconfigurational-4f transitions from excited state level $^6G_{5/2}$ to ground levels $^6H_{5/2}$, $^6H_{7/2}$ and $^6H_{9/2}$, respectively (Ning et al. 2019; Cui et al. 2020; Nandanwar et al. 2023h).

3.4 Chromatic properties

The colour of any light could well be represented as an (x, y) coordinate. The CIE chromaticity diagram of $KCaPO_4:Sm^{3+}$ Phosphor is shown in Fig. 8. The chromaticity coordinate of doping concentration was determined to be (0.602, 0.395) under the 403 nm excitation wavelength. The plotted CIE chromaticity diagram shows the coordinate located in the

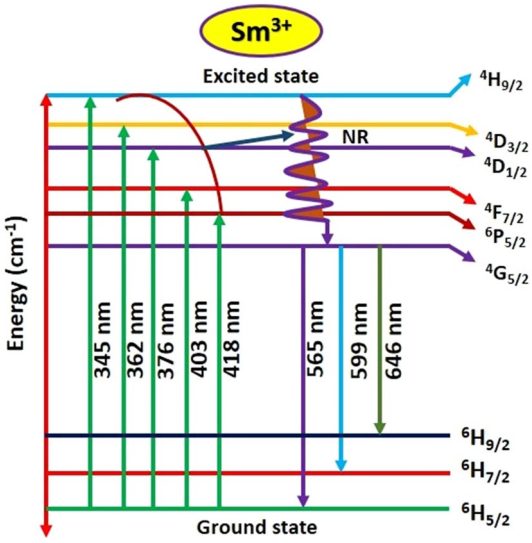

Fig. 7 Energy level diagram of the Sm^{3+} ions

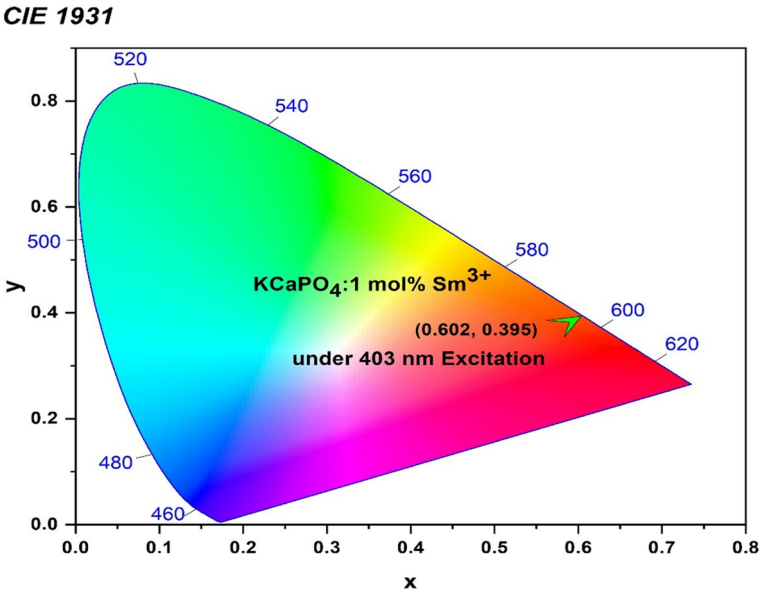

Fig. 8 CIE coordinate diagram of $KCaPO_4$:1 mol% Sm^{3+} phosphor

orange-red region (Ning et al. 2019; Baig et al. 2016; Yerojwar et al. 2022c). The color purity of the sample is determine by the following equations:

$$\text{Color purity} = \frac{\sqrt{(x-x_i)^2+(y-y_i)^2}}{\sqrt{(x_d-x_i)^2+(y_d-y_i)^2}} \times 100$$

where (x_i, y_i) represent the white illumination chromaticity coordinates, (x_d, y_d) is the dominant wavelength chromaticity coordinates and (x, y) of the $KCaPO_4$:1 mol% Sm^{3+} phosphor chromaticity coordinates (Zhao et al. 2019; Parauha et al. 2022). In this investigation, (x = 0.602, y = 0.395), (x_i = 0.310, y_i = 0.316) and (x_d = 0.613, y_d = 0.385). Therefore, the calculated color purity of synthesized $KCaPO_4$:1 mol% Sm^{3+} phosphor was around 96.90%. The obtained result of $KCaPO_4$:1 mol% Sm^{3+} phosphor has high color purity for promising applications in solid-state lighting under n-UV excitation.

4 Conclusion

In this study, the various concentration of novel $KCaPO_4$:Sm^{3+} phosphor was successfully first time prepared by wet chemical method. The XRD, morphological and photoluminescence properties were investigated. The $KCaPO_4$:Sm^{3+} phosphor excitation wavelength 403 nm was selected for the measurement of emission peak at yellow (565 nm), orange-red (599 nm) and red (646 nm). The optimal concentration of Sm^{3+} ions, as well as the concentration quenching effect, were obtained when x = 1 mol %. According to the experimental results and the theoretical calculation, it is identified that the q–q interaction plays the major role in the concentration quenching mechanism of $KCaPO_4$:Sm^{3+} phosphor. The

calculated CIE coordinates of $KCaPO_4$:1 mol% Sm^{3+} phosphor are (0.602, 0.395) and corresponding to orange-red region with a high color purity of about 96.90%. The results indicated that the produced $KCaPO_4:Sm^{3+}$ phosphor is a promising candidate for n-UV based solid-state lighting.

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Data availability No data was used for the research described in the article.

Declarations

Conflict of interest The authors declare that they do not have any known competing financial interests or personal relationships that could appear to have influenced the work reported in this paper.

Ethical approval Not applicable.

Consent to participate Not applicable.

Consent to publish Not applicable.

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