

Reinforced Optical Properties of Sm³⁺ Complexes with β -Hydroxyketone Ligand by Using Methylated Auxiliary Ligands

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Solution precipitation method was used to synthesize two avant-grade ternary complexes of Sm³⁺ ion with 2-hydroxy-4,6-dimethoxyacetophenone (HDMA) as prime ligand and 5,6-dimethyl-1,10-phenanthroline (dmphen) as auxillary ligand. Characterization of these complexes was accomplished by means of elemental analysis, ¹H NMR, FT-IR and photoluminescence spectroscopy. Emission spectra recorded at ambient temperature, displayed sharp emission bands at 564, 600 and 646 nm attributed to ⁴G_{5/2}→⁶H_j (j = 5/2, 7/2, 9/2) f-f transitions of metal ion. The decent decay time of luminescence and superior colour purity reported in complexes. The correlated colour temperature of these complexes established them as warm light source. The quantum yield was also evaluated for both complexes. HDMA alone could sensitize Sm³⁺ ion proficiently but the inclusion of auxillary ligands neocuproine (neo) or 5,6-dimethyl-1,10-phenanthroline (dmphen) resulted in reinforced optical properties. The results of emission spectra illustrated that these ternary complexes can be one of the most promising substances for fabrication of display devices.

Keywords: Sm³⁺ complexes, β -Hydroxyketone, Auxiliary ligands, Reinforced optical properties, Quantum yield.

INTRODUCTION

Rare earth metal complexes with organic ligands have gained remarkable concern in research environs because of its extraordinary usage in biomedical sensors and imaging devices [1,2], in OLEDs [3-8] and in optical amplification by doping in polymers [9,10]. The main distinctive optical characteristics of lanthanides complexes are immense purity of colour, narrow emission band width, large stoke shift, good thermal stability and milliseconds life time [11]. Albeit, direct excitation of lanthanides ions are not practically efficient because lanthanides ions are complexes with strong absorbing organic ligand, such as β -diketone, β -ketoesters, β -ketocarboxylic, which absorb strongly, transfer it to rare earth metal cations *via* antenna effect [12-14] and hence augmented metal cation's emission. 2-Hydroxy-4,6-dimethoxyacetophenone (HDMA) was chosen as the primary ligand to form complex with lanthanide ion leading to greater photoluminescence and good quantum yield.

Neutral chelating ligands like derivative of 1,10-phenanthroline have unique quality to replace the water molecules present

around the central metal ions [15-17]. Therefore, they can be used as auxillary ligands to synthesise the ternary lanthanide complexes and conform to the coordination number of lanthanide ions resulting in augmented luminescence, antimicrobial properties and antioxidant properties. Using 2-hydroxy-4,6-dimethoxyacetophenone (HDMA) as primary ligand and 2,9-dimethyl-1,10-phenanthroline (neo) and 5,6-dimethyl-1,10-phenanthroline (dmphen) as auxillary ligand, two ternary Sm³⁺ complexes **C1** and **C2** were synthesized as excellent photoluminescent material. Numerous techniques like elemental analysis, ¹H NMR and FT-IR spectroscopy used to analyze the composition of complexes. Detailed investigation was done to know photoluminescent behaviour of these complexes. The photoluminescent spectra exhibited that these ternary Sm³⁺ complexes can be one of the propitious substances for fabrication of display devices.

EXPERIMENTAL

The chemicals like 2,9-dimethyl-1,10-phenanthroline, 5,6-dimethyl-1,10-phenanthroline, Sm(NO₃)₃·6H₂O (99.9%),

phloroglucinol, $(\text{CH}_3)_2\text{SO}_4$, K_2CO_3 and xylene orange were procured from Sigma-Aldrich and thrice crystallized with ethanol before synthesizing the metal complexes. The elemental analysis performed using Thermoscientific Flash 2000 Elemental analyser. Estimation of percentage of Sm^{3+} was performed by complexometric titration with EDTA. The ^1H NMR spectra were scaled on Bruker Advance II 400 spectrometer using tetramethylsilane (TMS) as an internal reference (chemical shift in δ ppm). Infrared spectra were ascribed using Perkin Elmer spectrum 400 spectrophotometer from 4000-400 cm^{-1} in KBr pellets. The photoluminescent spectra for excitation and emission recorded at 400 V in solid state and at ambient temperature with the help of Hitachi F-7000 fluorescence spectrophotometer and observing $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ emission line of Sm^{3+} .

Synthesis of ligand: Ligand 2-hydroxy-4,6-dimethoxyacetophenone (HDMA) was synthesized using the reported method [18,19].

Synthesis of complexes

Sm(HDMA)₃·neo (C1): The synthesis of complex Sm(HDMA)₃·neo (C1) was accomplished *via* solution precipitation method. The ethanolic solution of 3 mmol HDMA ligand and 1 mmol neo was mixed with ethanolic solution of 1 mmol $\text{Sm}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$. Subsequently, the pH of mixture was fixed to 7, using M/20 NaOH solution with constant stirring till the white precipitates were formed. The precipitates were further stirred for 3 h at about 35 °C and then left to digest for 1 h. Subsequently, the precipitate was separated with the help of suction filter, cleansed with distilled water and finally with ethanol. Further these precipitates were dried in vacuum oven at 50 °C (Yield: 79%) Elemental analysis %: C, 60.87 (61.22); H, 6.14 (6.16); O, 32.58 (32.62).

Complex C1: m.f.: $\text{C}_{44}\text{H}_{45}\text{O}_{12}\text{N}_2\text{Sm}$, elemental analysis: C, 56.04 (55.97); H, 4.92 (4.80); N, 3.01 (2.96); O, 20.29 (20.33); Sm, 15.86 (15.92). IR (KBr, ν_{max} , cm^{-1}): 3008 (m), 2961 (m), 1618 (s), 1570 (s), 1540 (s), 1453 (m), 1406 (m), 1386 (s), 1362 (m), 1260 (s), 1225 (s), 1202 (s), 1160 (s), 1026 (s), 1079 (s), 966 (m), 853 (s), 832 (s), 695 (m), 596 (m), 535 (m), 449 (w); ^1H NMR (400 MHz, DMSO- d_6): δ 2.59 (s, 9H, CH_3), 2.69 (s, 9H, neo CH_3), 3.28 (s, 18H, OCH_3), 5.94 (s, 6H, Ar-H), 7.64 (d, 2H, neo), 8.17 (d, 2H, neo), 8.78 (d, 2H, neo).

Sm(HDMA)₃·dmphen (C2): Same procedure as employed in complex C1 was employed to synthesize the Sm(HDMA)₃·dmphen complex, apart from mixing dmphen (1 mmol) instead of neo as shown in Scheme-I.

Complex C2: White powder, yield: 90%. Elemental analysis of $\text{C}_{44}\text{H}_{45}\text{O}_{12}\text{N}_2\text{Sm}$: C, 56.06 (55.97); H, 4.84 (4.80); N, 2.74 (2.96); O, 20.41 (20.33); Sm, 15.78 (15.92). IR (KBr, ν_{max} , cm^{-1}): 2931 (w), 2832 (w), 2717 (m), 1616 (s), 1584 (s),

1532 (s), 1420 (m), 1365 (s), 1261 (s), 1225 (s), 1209 (s), 1158 (s), 1121 (m), 1076 (m), 963 (m), 865 (m), 834 (m), 776 (m), 712 (m), 595 (m), 534 (w), 472 (w), 428 (w); ^1H NMR (400 MHz, DMSO- d_6): δ 2.62 (s, 9H, CH_3), 2.71 (s, 9H, dmphen CH_3), 3.82 (s, 18H, OCH_3), 6.03 (s, 6H, Ar-H), 7.71 (d, 2H, dmphen), 8.52 (d, 2H, dmphen), 8.68 (d, 2H, dmphen).

RESULTS AND DISCUSSION

Stoichiometric analysis and solubility: The elemental analytical data for the HDMA, C1 and C2 revealed that the stoichiometry of ternary Sm^{3+} ion complexes to be 1:3:1 (Sm: HDMA:auxillary ligand). The results obtained confirm the molecular formulae of complexes as $\text{Sm}(\text{HDMA})_3 \cdot \text{neo}$ and $\text{Sm}(\text{HDMA})_3 \cdot \text{dmphen}$. Both these complexes are significantly stable at atmospheric condition and found to be fairly soluble in DMSO, chloroform and acetone but are sparingly soluble in hexane and benzene.

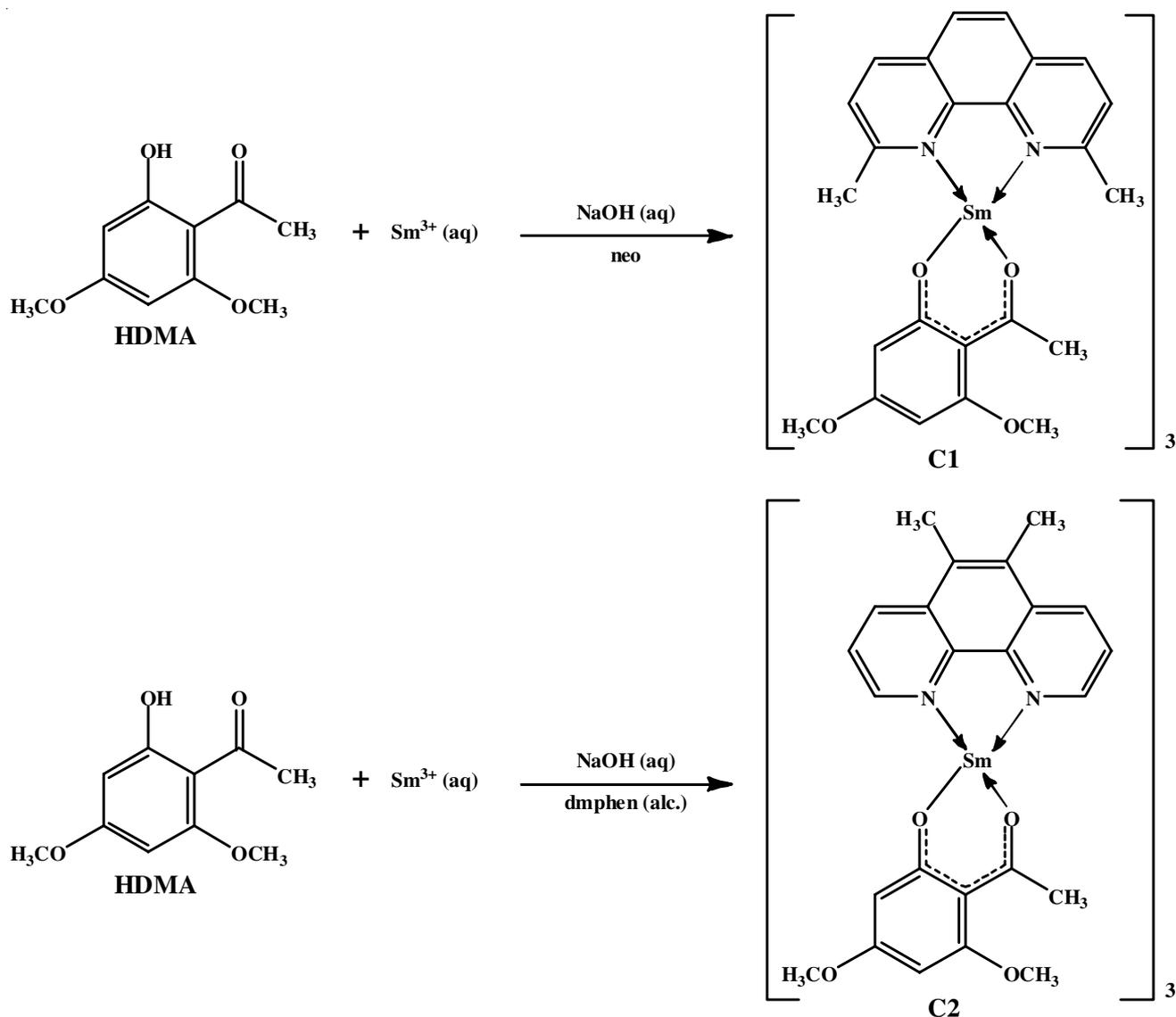
^1H NMR and FTIR spectral analysis: The bonding mode of complexes were studied through ^1H NMR and FT-IR spectra. Singlet at δ 13.84 ppm, appearing in the ^1H NMR spectrum of ligand HDMA because of phenolic proton, was vanished in the spectra of complexes C1 and C2 which indicated that HDMA is coordinately bonded to Sm^{3+} ion *via* oxygen atom of phenolic group. However, broadening and upfield-downfield shift of signal observed in spectra of complexes can be explained due to paramagnetic behaviour and significant anisotropy of Sm^{3+} ion.

The IR spectra of HDMA, C1 and C2 in KBr pellets recorded with Perkin-Elmer spectrum 400. The key IR bands are given in Table-1. In IR spectrum of uncoordinated ligand HDMA, a broad band at 3430 cm^{-1} is present which is assigned to stretching vibration of -OH [20-22], but it is vanished in the spectra of complexes. The band for Ph-O vibration of the ligand HDMA present at 1270 cm^{-1} blue shifted in both the complexes, 1259 cm^{-1} for complex C1 and 1261 cm^{-1} for complex C2, which demonstrate that the phenolic group is included in coordination with metal ion. The sharp stretching vibration band, attributed to C=O group at 1636 cm^{-1} is present in spectrum of free ligand and is shifted to 1618 cm^{-1} in C1 and 1609 cm^{-1} in C2 complexes, affirming the engagement of carbonyl group in chelation with the Sm^{3+} ion [23]. The strong absorption band at 1570 and 1584 cm^{-1} in spectra of complex C1 and C2 respectively, associated with the C=N stretching vibration, provided good evidence about the presence of nitrogen atom in auxillary ligands in the coordination sphere of Sm^{3+} ion [18,24]. The absorption bands present at 596 and 595 cm^{-1} associated with $\nu(\text{Sm-N})$ and at 449 and 428 cm^{-1} attributed to $\nu(\text{Sm-O})$ in spectra of complex C1 and C2, indicate that Sm^{3+} ion is bonded to the prime and auxillary

TABLE-1
CHARACTERISTIC IR BANDS (cm^{-1}) OF HDMA AND COMPLEXES C1 AND C2

Compound	$\nu(\text{O-H})$	$\nu(\text{C=O})$	$\nu(\text{C=N})$	$\nu(\text{C=C})$	$\nu(\text{Ph-O})$	$\nu(\text{Sm-N})$	$\nu(\text{Sm-O})$
HDMA	3430 b	1636 s	–	1538 s	1270 m	–	–
C1	–	1618 s	1570 m	1538 s	1259 m	596 w	449 w
C2	–	1609 s	1584 m	1532 m	1261 m	595 w	428 w

s = sharp, m = medium, w = weak, b = broad



Scheme-I: Synthetic route of complexes **C1** and **C2**

ligand *via* O and N atom, respectively. Finally, it can be inferred from the FT-IR and ^1H NMR spectra of the free HDMA and its corresponding Sm^{3+} complexes **C1-C2**, that the coordination of Sm^{3+} with ligand is through the O atoms of phenolic and carbonyl group of HDMA whereas through N atoms for corresponding auxiliary ligand.

Photoluminescent properties: The excitation spectra of the prime ligand HDMA and its Sm^{3+} complexes (**C1**, **C2**) were recorded by monitoring $^4\text{G}_{5/2} \rightarrow ^6\text{H}_{7/2}$ transition (Fig. 1). In spectrum of HDMA, a broad band ranges from 260 to 435 nm is present, but the excitation spectra of complexes is extended over a comprehensive range of 250 to 450 nm, with the peaks centred at 374 nm and 403 nm. Electronic transitions associated with the peaks are $^6\text{H}_{5/2} \rightarrow ^4\text{D}_{1/2} + ^6\text{P}_{7/2}$ and $^6\text{H}_{5/2} \rightarrow ^4\text{F}_{7/2}$, respectively [25]. Peak at 373 nm has high excitation intensity as compared to the other peak and so this excitation intensity was selected for measuring emission spectra. The strong peak at 374 nm illustrates that the complexes are effectively excited in the near ultra violet region.

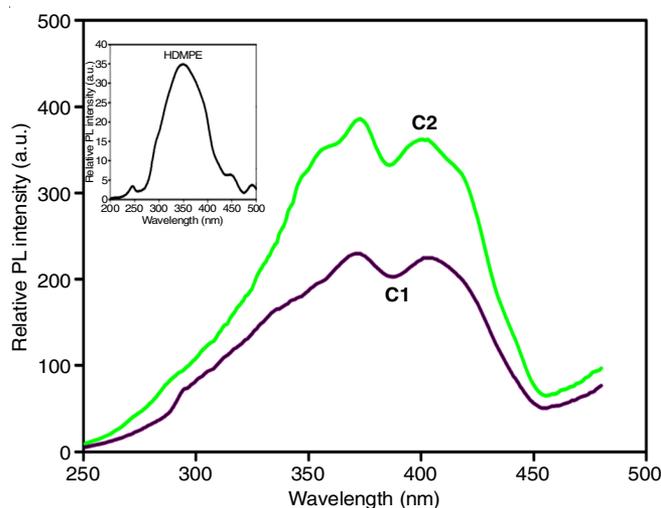


Fig. 1. Photoluminescence excitation spectra of Sm^{3+} complexes **C1-C2**, monitored at $\lambda_{\text{em}} = 600$ nm in solid state at room temperature and inset shows the excitation spectrum of ligand HDMA

Fig. 2 demonstrates the solid state emission spectra of complexes and ligand which displayed three emission peaks. Peaks at 564, 600 and 646 nm attributed to ${}^4G_{5/2} \rightarrow {}^6H_j$ where $j = 5/2, 7/2, 9/2$ $f-f$ transitions respectively [26-28], which were obtained by fixing excitation wavelength at 374 nm at ambient temperature (Table-2). Out of these three transitions, ${}^4G_{5/2} \rightarrow {}^6H_{5/2}$ is magnetic dipole transition and ${}^4G_{5/2} \rightarrow {}^6H_{9/2}$ is electric dipole transition whereas ${}^4G_{5/2} \rightarrow {}^6H_{7/2}$ is mixed transition. The magnetic-electric dipole transition ${}^4G_{5/2} \rightarrow {}^6H_{7/2}$ is strongest in ternary complexes **C1** and **C2** while the electric dipole ${}^4G_{5/2} \rightarrow {}^6H_{9/2}$ transition is subsidiary in most of Sm^{3+} complexes [29-31].

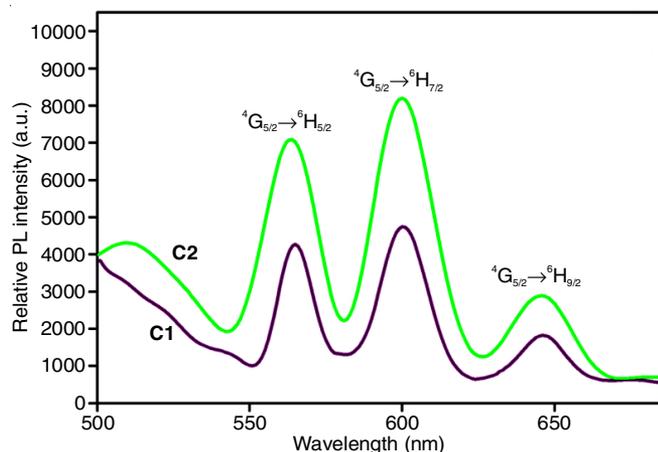


Fig. 2. Photoluminescence emission spectra of Sm^{3+} complexes **C1-C2** in solid state at room temperature, monitored at $\lambda_{ex} = 374$ nm

TABLE-2
PHOTOLUMINESCENCE DATA OF $Sm(III)$
COMPLEXES **C1-C2** IN SOLID STATE

Complexes	λ_{ex} (nm)	λ_{em} (nm)	Assignment	Life time (ms)	Quantum yield (%)
C1	373	564	${}^4G_{5/2} \rightarrow {}^6H_{5/2}$	0.966	29.81
		600	${}^4G_{5/2} \rightarrow {}^6H_{7/2}$		
		646	${}^4G_{5/2} \rightarrow {}^6H_{9/2}$		
C2	373	564	${}^4G_{5/2} \rightarrow {}^6H_{5/2}$	0.769	23.73
		600	${}^4G_{5/2} \rightarrow {}^6H_{7/2}$		
		646	${}^4G_{5/2} \rightarrow {}^6H_{9/2}$		

The photoluminescence intensity of ternary complexes was found to be more reinforced in comparison to the binary complexes, because in the ternary complexes OH groups replaced from the inner coordination sphere of Sm^{3+} ion. The replacement of water molecules by aromatic N-donors auxiliary ligands augmented the photoluminescence intensity and stability of complexes [23,32]. Further, it was observed that the auxiliary ligands in the ternary complexes augmented the photoluminescence intensity consequent to the coordination with primary ligand and auxiliary ligand and thus facilitates effective intramolecular transfer of energy. Complexes **C1** and **C2** exhibited low intensity ratio of the electric dipole to the magnetic dipole transition revealing the Sm^{3+} ion holds site symmetry with an inversion centre.

Fig. 3 depicts the photoluminescence decay curves for complexes **C1** and **C2** at $\lambda_{em} = 600$ nm and $\lambda_{ex} = 374$ nm at the ambient temperature. The decay curves of luminescent ternary

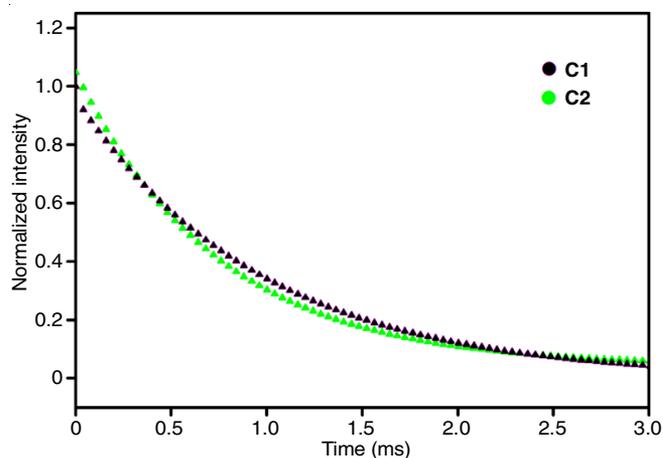


Fig. 3. Luminescence decay curves for complexes **C1-C2** in solid state at room temperature, monitored at $\lambda_{ex} = 374$ nm and $\lambda_{em} = 600$ nm

complexes **C1** and **C2** comply with single exponential behaviour and can be described by the following equation:

$$I = I_0 e^{-t/\tau} \quad (1)$$

where τ is the radioactive decay time, I is the luminescence intensities at time t and I_0 is the luminescence intensity at any time ' t '. The decay time values obtained for complexes were found to be 0.966 ms (**C1**) and 0.769 ms (**C2**) as tabulated in Table-3.

TABLE-3
CHROMATICITY COORDINATES, COLOUR PURITY
AND CORRELATED COLOUR TEMPERATURE OF
 Sm^{3+} COMPLEXES OF HDMA, **C1** AND **C2**

Complexes	CIE coordinates		Colour purity (%)	CCT (K)
	x	y		
C1	0.5312	0.4578	93.61	2237.633
C2	0.5272	0.4605	95.56	2290.969

Quantum yield is obtained for complexes to further delve into the luminescent behaviour of complexes. The quantum yield (τ) is the ratio of photons emitted to the photons absorbed and is numerically calculated by the following equation [33]:

$$\phi = \frac{\tau}{\tau_{rad}} \times 100 \quad (2)$$

where, τ is the observed life time and τ_{rad} is the radiative life time. The value of τ_{rad} of transition ${}^6G_{5/2}$ manifold for Sm^{3+} is 3.24 ms [34]. The quantum yield fetched for complexes **C1** and **C2** are 29.81 and 23.73%, respectively (Table-3). Decent quantum yield of complexes can be explicated because of effective transfer of energy and less nonradiative decay.

Colour analysis: The colour emitted by the complexes was further analyzed using CIE (Commission Internationale de l'Eclairage) chromaticity coordinate diagram. The colour coordinates (x,y) values procured from emission data and MATLAB software of the complexes are 0.5312, 0.4578 for **C1** and 0.5272, 0.4605 for **C2** (Table-3). The complexes were found to be present in dark orange region of colour gamut space (Fig. 4), suggesting promising substances for fabrication of display devices.

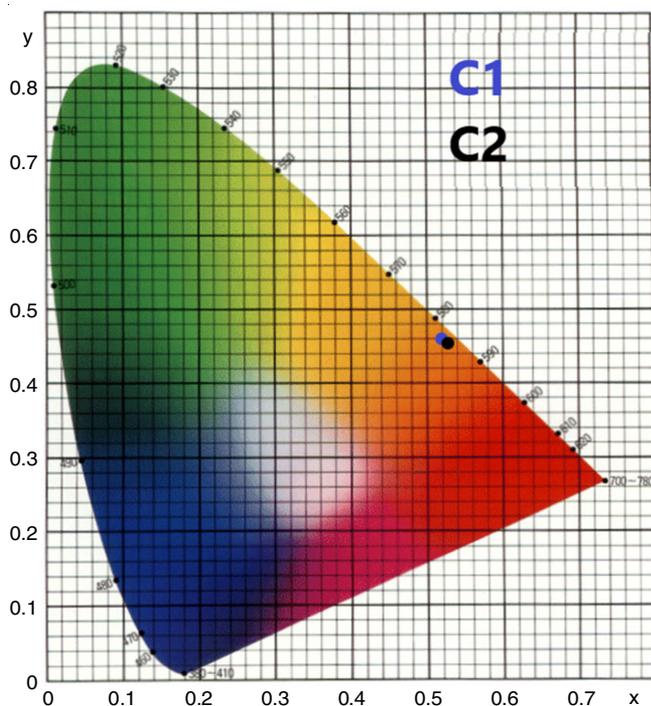


Fig. 4. CIE coordinate diagram of Sm(III) complexes C1-C2

Colour purity percentage (CP%) of complexes was estimated in reference to CIE illuminated coordinates (x_i , y_i). Colour purity of any sample is the weighted average of its colour coordinates of emission and dominant wavelength coordinates (x_d , y_d) with the respective illuminated coordinates of white light ($x_i = 0.33$, $y_i = 0.33$). The colour purity of the Sm³⁺ complexes was evaluated by means of eqn. 3 [35] and the values of obtained are presented in Table-3, which reveal the superior colour purity of the synthesized complexes.

$$CP (\%) = \frac{\sqrt{(x - x_i)^2 + (y - y_i)^2}}{\sqrt{(x_d - x_i)^2 + (y_d - y_i)^2}} \times 100 \quad (3)$$

To analyse the colour approach of emitted light, correlated colour temperature (CCT) values of complexes C1-C2 are also determined numerically using following mathematical expression [36]:

$$CCT = -449n^3 + 3525n^2 - 6823n + 5520.33 \quad (4)$$

$$n = \frac{x - x_e}{y - y_e} \quad (5)$$

In aforesaid equation x , y are CIE coordinates and x_e , y_e are the colour epicentres of the corresponding axis. The values of x_e and y_e are given as 0.3320 and 0.1858, respectively. The CCT values procured from eqn. 4 comes out to be 2237.63 and 2290.96 K for complexes C1 and C2, respectively. Since the values are < 3200 K so these complexes are categorized as warm light source [37,38] and hence can be suitable for lights in home appliances, restaurants and lounges.

Conclusion

Two avant grade ternary Sm³⁺ complexes Sm(HDMA)₃neo (C1), Sm(HDMA)₃dmphen (C2) were synthesized and

characterized by elemental analysis, FT-IR and ¹H NMR spectroscopy. The IR and NMR spectra of the uncoordinated ligand (HDMA) exhibited significant variation when contrasted with complexes indicating the bonding aspects of complexes. Besides, the endorsed coordination surrounding is strongly supporting the sensitization process and the luminescence of Sm³⁺ ion, which leads to efficient and dark orange emission. Moreover, inclusion of auxillary ligands reinforced photoluminescence properties and decay time. In conclusion, the complexes C1 and C2 had outstanding luminescent properties, thus can be encouraging substances for display devices.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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