Synthesis and spectroscopic characterization of lanthanide complexes derived from 9,10-phenanthrenequinone and Schiff base ligands containing N, O donor atoms

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A series of mixed ligand lanthanide complexes of general formula $[Ln(PhenQ)(L_{1-3}) (NO_3)_2(H_2O)_n]$ [where n = 0, Ln = Yb(III); n = 1, Ln = Dy(III); and n = 2, Ln = Nd(III)] have been derived from 9,10-phenanthrenequinone (PhenQ) and Schiff bases $(L_{1-3}H)$ [where, $L_1H = (E)$ -2-methoxy-6-((thiazol-2-ylimino) methyl) phenol, $L_2H = (E)$ -4-(2-hydroxy-3-ethoxybenzylidineamino) benzoic acid and $L_3H = 2$ -((p-tolylimino) methyl) phenol] containing N, O donor atoms. These metal complexes have been characterized by elemental analysis, infrared, UV-Vis, Thermogravimetric analysis, Powder X-ray diffraction and ESI-Mass spectrometry. Surface morphology of the synthesized complexes has been analysed by SEM-EDX and they appear to have stone-like surface morphology (rough, with indefinite shape). The metal complexes of Nd and Dy have been found to be ten coordinated, whereas metal complex of Yb is found to be eight coordinated which has tentatively been proposed by spectroscopic techniques.

Keywords: Lanthanide complexes, Schiff bases, Powder X-ray diffraction, Thermogravimetric analysis

Lanthanide complexes with N, O donor Schiff bases and organic ligands¹⁻⁹ are of great interest due to their potential biological, clinical, analytical and industrial applications¹⁰⁻¹⁴. Lanthanide ions in their trivalent state draw considerable attention due to their complexation with strong chelating ligands containing electronegative donor atoms¹⁵ well-defined spectroscopic and magnetic properties¹⁶⁻²⁰. lanthanide complexes are Nowadays. being extensively studied for magnetic and luminescent properties for various purposes²¹. In the recent years, lanthanide complexes containing quinones and quinoids have drawn considerable attention for toxicological studies because of their distinctive roles in the fields of organic and biochemistry²²⁻²⁵, whereas 9,10-phenanthrenequinone found in the particulate segment of air effluent samples including ambient air and diesel exhaust particles²⁶ generates interest from the toxicological stand point because of its quinone-like chemistry²⁷. In consideration to the above facts, the present investigation deals with synthesis, characterization, and thermal studies of four Ln(III) (where Ln = Nd, Dy, Yb) complexes containing PhenQ and Schiff bases.

Materials and Methods

The Nd(NO₃)₃.6H₂O,Dy(NO₃)₃.5H₂O, Yb(NO₃)₃.5H₂O, 9,10-phenanthrequinone, *p*-toluidine, salicylaldehyde, *o*-vanillin, 4-aminobenzoic acid and 2-aminothiazole were purchased from Sigma Aldrich and used without further purification. AR grade solvents were used for synthesis and recrystallization.

Physical measurements

Melting points of the ligands and lanthanide(III) complexes were recorded on MAC digital melting point apparatus. The FT-IR spectra (4000–400 cm⁻¹) were recorded using KBr pellets on a PerkinElmer 1000 FT-IR spectrophotometer. The elemental (C, H and N) analysis was performed on CHN-932 PerkinElmer 7300 DV elemental analysers. Powder X-ray diffraction (PXRD) was recorded on X' Pert Pro XRD. HRMS were recorded in ESI mode on Xevo G2-S Q Tof. SEM-EDAX was recorded on JEOL, Japan (JFC 1600, Auto Fine Coater). Thermogravimetric analyses (TGA) and differential thermal analyses (DTA) were performed on a STA

[#] Deceased since November 2018

6000 PerkinElmer instrument under inert N_2 atmosphere. UV-Vis spectra were recorded in DMSO solution, at room temperature and wavelength was recorded in nm using a UV-1800 Shimadzu UV spectrophotometer.

Synthesis of Schiff base ligands $(L_1H - L_3H)$

(*E*)-2-methoxy-6-((thiazol-2-ylimino) methyl) phenol (L_1H) was prepared (Scheme 1) in methanol by condensation of 2-aminothiazole and *o*-vanillin in 1:1 molar ratio. Vanillin (1.519 g, 9.98 mmol) and 2-aminothiazole (1.000 g, 9.98 mmol) were mixed in methanol (~ 40 cm³) and refluxed for ~7 h to afford yellow solid product, which was washed with cold methanol and air-dried. Recrystallization from hot methanol gave analytically pure yellow solid. Yield: 70%, m.pt: 110 °C; Anal. (%): Found: C, 55.92; H, 4.01; N, 11.09. Calcd: C, 56.39; H, 4.30; N, 11.96.

A similar procedure was adopted to prepare Schiff base ligands (L_2H-L_3H).

(*E*)-4-(2-hydroxy-3-methoxybenzylidineamino) benzoic acid (L_2H): Yield: 76%. M.pt: 240 °C; Anal. (%): Found: C, 65.99; H, 4.40; N, 4.42. calcd: C, 66.41; H, 4.83; N, 5.16.

2-((p-tolylimino) methyl) phenol²⁸ (**L**₃**H**): Yield: 83%, m.pt: 61 °C; Anal. (%): Found: C, 79.23; H, 6.11; N, 6.35. calcd: C, 79.59; H, 6.20; N, 6.63.

Synthesis of complexes (1-4)

All lanthanide(III) complexes (1-4) were synthesized by using the same procedure. [(PhenQ)(L₃)Yb(NO₃)₂] (1) was synthesized as follows: To a methanolic solution of PhenQ (0.463 g, 2.22 mmol), methanolic solution of Yb(NO₃)₃.5H₂O (1.000 g, 2.22 mmol) was added drop wise with constant stirring. To this solution, potassium salt of Schiff base ligand, L₃K (0.470 g, 2.22 mmol) (prepared by dissolving an equimolar amount of potassium hydroxide (0.124 g, 2.21 mmol) and Schiff base (0.470 g, 2.22 mmol) with constant stirring ~ 2 h in MeOH (30 mL)) was added drop wise with constant stirring in 1:1 molar ratio and the resulting mixture was refluxed for ~24 h. The precipitated KNO₃ was removed by filtration. The filtrate was concentrated by removing the excess solvent by distillation. The product was dried under reduced pressure and recrystallized by methanol/chloroform mixed solvent.

[(PhenQ)(L₃)Yb(NO₃)₂] (1): Yield: 62%, m.pt: 193–198 °C; Anal. (%): Found for $C_{28}H_{20}N_3O_9Yb$: C, 46.67; H, 2.42; N, 5.37. calcd: C, 47.00; H, 2.82; N, 5.87. TOF-MS (*m/z*): Found (calcd) 716.17 (715.51).

[(PhenQ)(L₁)Dy(NO₃)₂(H₂O)] (**2**): Yield: 72%, m.pt: 287–195 °C; Anal. (%): Found for $C_{25}H_{19}DyN_4O_{11}S$: C, 40. 15; H, 2.48; N, 7.43. calcd: C, 40.25; H, 2.57; N, 7.51. TOF-MS (*m/z*): Found (calcd) 745.41 (746.00).

[(L₂)(PhenQ)Nd(NO₃)₂(H₂O)₂] (**3**): Yield: 65%, m.pt: 197 °C; Anal. (%): Found for $C_{29}H_{24}N_3NdO_{14}$: C, 44.39; H, 3.01; N, 5.27. calcd: C, 44.50; H, 3.09; N, 5.37. TOF-MS (m/z): Found (calcd) 782.69 (782.75).



[(L₃)(PhenQ)Nd(NO₃)₂(H₂O)₂] (4): Yield: 69%, m.pt: 140 °C; Anal. (%): Found for $C_{28}H_{24}N_3NdO_{11}$: C, 46.41; H, 3.28; N, 5.71. calcd: C, 46.53; H, 3.35; N, 5.81. TOF-MS (*m/z*): Found (calcd) 722.52 (722.74).

Results and Discussion

Equimolar amounts of lanthanide(III) nitrate, potassium salts of Schiff bases and PhenQ in 1:1:1 stoichiometric ratio in MeOH, afforded lanthanide(III) complexes (Scheme 2) of type $[Ln(phenQ)(L_{1-3})(NO_3)_2(H_2O)_n]$ (1-4). All these complexes are coloured solids, soluble in DMF, chloroform, DCM, acetonitrile, DMSO and slightly soluble in methanol. These lanthanide complexes are stable in air.

Infrared spectral studies

The important IR spectral bands of the Schiff bases $(L_{1-3}H)$ and their lanthanide complexes are given in Table 1. The band observed in the region 3160–3460 cm⁻¹ due to v(O-H) are present in Schiff bases $(L_{1-3}H)$, whereas these bands are absent in the lanthanide complexes. In comparison with Schiff



where n = 0, Ln = Yb; n = 1, Ln = Dy; and n = 2, Ln = Nd



Table 1 — Major infrared spectral data for Schiff bases $L_{1-3}H$ and their Ln(III) complexes										
Compound	$v(H_2O)$	v(OH)	ν(C=N)	v(ArO)	$\nu(NO_3)$			v(M-O)	v(M-N)	
					ν_1	ν_2	v ₃	$v_1 v_3$	_	
L ₁ H		3237	1631	1251	-	-	-	-	-	-
L ₂ H		3160	1645	1251	-	-	-	-	-	-
L ₃ H		3458	1616	1268	-	-	-	-	-	-
[(PhenQ)(L ₃)Yb(NO ₃) ₂]	3250	-	1645	1278	1489	823	1380	109	510	462
$[(PhenQ)(L_1)Dy (NO_3)_2(H_2O)]$	3360	-	1638	1238	1441	829	1325	116	530	435
[(PhenQ) (L ₂)Nd (NO ₃) ₂ (H ₂ O) ₂]	3427	-	1653	1258	1441	877	1387	54	530	435
[(PhenQ) (L ₃)Nd (NO ₃) ₂ (H ₂ O) ₂]	3366	-	1611	1278	1496	815	1360	136	516	428

bases, the Ln(III) complexes exhibit broad band around 3250–3450 cm⁻¹ which are attributed to the υ (O-H) stretching frequencies and shows the existence of coordinated water²⁹. The stretching frequency due to $v(H_2O)$ are observed in the region 3250-3450 cm⁻¹ in metal complexes and are absent in ligands. The stretching frequency due to v(C=N) are observed in the region 1610–1660 cm⁻¹. The variation in the band region from ligand to its complexes favours the formation of their metal complexes. A strong vibration appeared at 1626–1635 cm⁻¹ in the complexes corresponding to quinone carbonyls v(C=O) bonding with metals³⁰. The band observed in the region 1250–1280 cm⁻¹ is due to v(Ar-O) and the band also shows variation after complexation. The band observed the region 510-530 cm⁻¹ and 425-465 cm⁻¹ is due to v(M-O) and v(M-N) which were absent in the ligands. The characteristic frequencies of the coordinating nitrate groups appear at about 1441–1496 cm⁻¹ (v_1) and are associated with asymmetric stretching, 815–877 cm⁻¹(v_2) associated with symmetric stretching, 1325C1387 cm⁻¹(υ_3) and the difference between two highest frequency bands $|\Delta v = v_1 - v_3|$ is 54–136 cm⁻¹, indicating that nitrate ions in the solid complex coordinate to Ln(III) metal ions as bidentate ligands³¹⁻³². (Supplementary Data, Fig. S1).

Electronic spectra

The UV-Vis absorption spectra of the Schiff base ligands (L_1H-L_3H) and their Ln(III) complexes have been recorded in DMSO solution in the range of 250–900 nm at room temperature. The UV-Vis spectra of these Schiff base ligand L_3H and Yb(III) complex are shown in Fig. 1.

The variation in the absorption spectrum of Schiff base ligands and their Ln(III) complexes indicate the formation of complexes. In the absorption spectrum



Fig. 1 — The UV-Vis absorption spectrum of ligand (L_3H) and $[(PhenQ)(L_3)Yb(NO_3)_2]$ (1) in DMSO at room temperature.

of ligands a π - π * transition assigned to -C=N (chromophore) is observed³³⁻³⁴ at ~324 nm. These bands shifted towards higher intensity as well as wavelength after lanthanide gets coordinated in complex 1, whereas in complexes 2 and 3 the bands are shifted towards lower wavelength. The intensity band also gets shifted at lower range as observed in complex 3. Thus the UV–Vis spectra show that the intensities of λ_{max} for lanthanide complexes are different than those of the ligand, with a moderate change in their wave number (Supplementary Data, Fig. S2).

Mass spectral studies

The TOF-MS ES⁺ spectral data for the complexes 1-4 were recorded and different fragmentation patterns with m/z have been suggested. All the m/zvalues are assigned in $[M]^+$ molecular ion form. In mass spectra of the complexes 1-4, most of the fragment ions were observed in the form of group of peaks due to the presence of various isotopes of lanthanide metals. In the spectra of the complexes (1-4), the molecular ion peaks are observed at m/z716.1705 (calcd m/z 715.5100), 745.4131 (calcd m/z 746.0032), 782.6955 (calcd m/z 782.7523), 722.5233 (calcd m/z 722.7432) due to $[C_{28}H_{20}N_3O_9Yb]$ (1), $[C_{25}H_{19}DyN_4O_{11}S]$ (2), $[C_{29}H_{24}N_3NdO_{14}]$ (3) and $[C_{28}H_{24}N_3NdO_{11}]$ (4), respectively (Supplementary Data, Fig. S3). In these spectra, the base peak observed due to the fragment $[C_{14}H_8O_2]^{\bullet^+}$ at m/z 208.1741 (1), $[C_{14}H_{10}DyO_3)]^{\bullet^+}$ at m/z 389.0715 (calcd 388.7274) (2), $[(C_{14}H_8O_2)]^+$ at m/z 209.7512 (3) and $[C_{14}H_{13}NO]^+$ at m/z 211.7503 (4). In complex (1) other important peaks were also observed at m/z 596.6681 (calcd594.5033) and 381.4303 (calcd 383.2912) due to formation of different fragments $[C_{28}H_{20}NO_3Yb]^+$ and $[C_{14}H_{12}NOYb]^+$ respectively. In $[(PhenQ)(L_1)Dy(NO_3)_2(H_2O)]$ complex (2) two another peaks observed at m/z 623.9671 (calcd 621.9938) and 209.9511 (calcd 208.216) were due to formation of different fragments $[(C_{25}H_{19}DyN_2O_5S)]^+$ and $[C_{14}H_8O_2]^{\bullet+}$ respectively. In complex (3) peaks observed at *m/z* 657.1523 (calcd 658.7428) and 451.2438 (calcd 450.5306) were due to formation of fragments $[(C_{29}H_{24}NNdO_8)]^{\bullet^+}$ and $[(C_{15}H_{16}NNdO_6)]^{\bullet^+}$ respectively, whereas in complex (4) peaks observed at *m/z* 563.7938 (calcd 562.7033) were and 353.2874 (calcd 352.4521) were due to formation of different fragments $[(C_{28}H_{20}NNdO_3)]^+$ and $[(C_{14}H_8NdO_2)]^{\bullet^+}$ respectively.

SEM-EDX analysis

Scanning electron microscopy (SEM) is used to discuss the surface morphology of the lanthanide complexes by scanning the surface with focused beam of electrons. The SEM images have a fluffy and fleecy appearance for all the complexes, excluding the surface of the complex (1) (Fig. 2). The complex (1)resembles stone like surface morphology. The chemical composition of Ln(III) complexes were determined using energy dispersive X-ray spectroscopy (EDX). It is a technique used for elemental analysis and chemical characterization of samples. EDX of the complexes were also recorded which gives the information about the presence of different lanthanides (Yb, Nd, Dy), carbon, oxygen elements (Supplementary Data, Fig. S4). The EDX profile of Nd(III), Yb(III) and Dy(III) complexes



Fig. 2 — SEM images of (a) Nd(III) complex, (3) (b) Yb(III) complex (1), (c) Nd(III) complex (4), and, (d) Dy(III) complex (2).

show peaks of C, O and Ln(III) elements which are supporting the proposed structure.

Thermogravimetric studies

The thermal behaviour of Ln(III) complexes (2-4) under inert N_2 atmosphere conditions were investigated by TGA-DTA techniques. There is a correspondence quantitative between all the curves, which exhibits a weight loss with temperature. The decomposition products have been identified on the basis of analysis and mass spectral data (Supplementary Data, Fig. S5). The $[(L_2)(PhenQ)Nd(NO_3)_2(H_2O)_2]$ (3) complex undergoes two step decomposition pattern and DTA of this complex shows two endothermic decomposition patterns. The first step of decomposition occurs within the temperature range 83-90 °C corresponding to the mass loss 12.79% (calcd 12.53%) by decomposition of two H₂O and one NO₃ unit. The second step of decomposition occurs at the temperature range (290-480 °C) with mass loss 42.92% due to $[C_{21}H_{17}NO_3]$. The mass of final residue corresponds to stable Nd₂O₃ + C. TGA analysis of $[(L_3)(PhenQ)Nd(NO_3)_2(H_2O)_2]$ (4) complex confirmed the presence of water moiety

in this complex and DTA shows endothermic decomposition peaks. The thermogram of this complex reveals that two H₂O and one NO₃ molecule decomposed around 30-90 °C with a weight loss 14.02% (calcd 13.57%). After continuous weight loss of 59.41% by removal of organic moiety $[C_{28}H_{21}N_2O_3]$ the mass of final stable residue correspond to $Nd_2O_3/2 + 2C$ at $(788.39 \ ^{\circ}C)^{35}$. The $[(PhenO)(L_1)Dv(NO_3)_2(H_2O)]$ (2) complex undergoes two step, endothermic decomposition pattern. The first decomposition occurs at 40-143 °C with mass loss 18.11% (calcd 19.03%) due to the removal of two NO₃ and one H₂O molecule. The second decomposition occurs at temperature range 190-600 °C with mass loss 58.44% (calcd 59.28%) due to the removal of organic components. The mass of final residue correspond to $Dy_2O_3/2$ at temperature 699 °C.

Powder XRD studies

Powder X-ray diffraction (PXRD) is a widely used technique to characterize crystalline solid compounds. The PXRD spectrum of complexes (1-4) was recorded over the $2\theta = 0-80^{\circ}$ range and wavelength of 1.54184Å (Fig. 3). There was no



Fig. 3 — PXRD spectra of (a) Yb(III) complex, (1) (b) Dy(III) complex (2), (c) Nd(III) complex (3), and, (d) Nd(III) complex (4).

Table 2 — PXRD analysis of Nd(III) (3) and Nd(III) (4) complexes						
Unit cell Parameters		$[(L_2)(PhenQ)Nd(NO_3)_2(H_2O)_2]$ (3)	$[(L_3)(PhenQ)Nd(NO_3)_2(H_2O)_2]$ (4)			
Lattice constant	a(Å)	6.4	18.5			
	b(Å)	6.4	18.5			
	c(Å)	3.7	10.4			
Unit cell volume $V(\text{\AA})^3$		151.6	3559.4			
Edge length		$a = b \neq c$	$a = b \neq c$			
Inter axial angle		$\alpha = \beta = 90, \gamma = 120$	$\alpha = \beta = 90, \gamma = 120$			
Crystal system		Hexagonal	Hexagonal			
2θ (degree)		40.0	16.5			
d-value(Å)		2.2	5.3			

Table 3 — Analysis of miller indices of Nd(III) complexes using ICPDS data

		301 D5 du	u					
Nd(III) (3) complex								
20	D (calc.)	D (obs.)	h	k	1			
29.001	2.983	3.075	1	0	1			
40.012	2.173	2.251	2	0	1			
49.483	1.792	1.840	2	1	1			
65.923	1.002	1.145	3	1	1			
Nd(III) (4) complex								
20	D (calc.)	D (obs.)	h	k	1			
11.046	7.819	8.004	2	0	0			
16.570	5.269	5.345	3	0	0			
19.862	4.359	4.465	3	1	0			
23.790	3.597	3.736	4	0	1			

well-defined crystalline peak in the spectrum of Yb(III) **1** and Dy(III) **2** complexes. It thus indicates that these complexes are amorphous in nature. Whereas $[(L_2)(PhenQ)Nd(NO_3)_2(H_2O)_2]$ (**3**) and $[(L_3)(PhenQ)Nd(NO_3)_2(H_2O)_2]$ (**4**) exhibit X-ray diffraction patterns consistent with the crystalline nature of the material. The particle size is calculated by using the Debye formula as given below:

$D = 0.91\lambda/\beta \cos\theta$

where, constant 0.91 is the shape factor, λ is the wavelength of Cu-Ka radiation, θ is the Bragg's diffraction angle and β is the full width at half maximum (FWHM). The above indexing method also gives miller indices (*hkl*), unit cell parameters and unit cell volume. All the peaks were compared with the JCPDS. The value of these parameters of the respective Nd(III) (**3**) and Nd(III) (**4**) complexes are shown in Tables 2 and 3.

Conclusions

The Nd(III), Yb(III) and Dy(III) complexes with Schiff base ligand derived from salicylaldehyde, 2-aminothiazole,4-amino benzoic acid, *o*-vanillin, *p*-toluidine and PhenQ were synthesized and characterized by elemental analysis, Mass, IR, UV-Vis, TGA and PXRD. The Nd(III) ions complexes (**3**, **4**) are ten coordinated by nine O atoms: four of which belong to nitrate ions, two to PhenQ and remaining to water and Schiff base ligands, and, one by N atom. Dy(III) complex is nine coordinated by eight O atoms, four of which come from nitrate ions, two from PhenQ, remaining from water and Schiff base ligand and one by N atom, whereas Yb(III) ions are eight coordinated by seven O atoms, four of which come from nitrate ions, two from PhenQ, one from Schiff base ligand and one by N atoms.

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Supplementary Data

Supplementary data associated with this article are available in the electronic form at http://www.niscair.res.in/jinfo/ijca/IJCA_58A(04)446-453_SupplData.pdf.

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