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Research Article

Synthesis, characterization and biological activity of Mixed ligand Lanthanum (III) complexes, Derived from 1-(2-pyridylazo)-2-naphthol and secondary ligands

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Abstract:

This study involved the preparation of two lanthanum (III) complexes using 1-(2-pyridylazo)-2-naphthol (PAN) as the primary ligand, combined with the secondary ligands 1,10-phenanthroline (Phen) and 8-hydroxyquinoline (8HQ). The complexes were synthesized by controlling the reaction medium with a buffer solution. The prepared complexes were characterized by spectroscopic analyses, including Fourier-transform infrared spectroscopy (FT-IR), UV-Vis spectroscopy, and proton nuclear magnetic resonance (^{1}H-NMR), as well as by elemental analysis (CHN) and molar conductivity measurements. In the [La(PAN)$_2$(NO$_3$)] complex, the PAN ligand coordinates as a tridentate ligand through the azo nitrogen, the pyridine-ring nitrogen, and the phenolic oxygen atom, while the nitrate group behaves as a bidentate ligand. Thus, the coordination number of the central atom is eight. In the mixed-ligand complexes, the secondary ligand Phen coordinates as a bidentate ligand through its two nitrogen donor atoms (N,N). In the [La(PAN)$_2$(8HQ)] complex, the 8HQ ligand coordinates as a bidentate ligand via its nitrogen and oxygen donor atoms (N,O). Based on the spectroscopic data, the primary ligand PAN behaves consistently as a tridentate ligand (N,N,O). The biological activity of these complexes was studied using the agar diffusion method. The study revealed that the mixed-ligand complexes exhibited enhanced biological activity, particularly the [La(PAN)$_2$(8HQ)] complex.

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Introduction

The most extensive series in the periodic table consists of lanthanides [1]. The atomic numbers 58 to 71, indicate that these elements predominantly exhibit a 3^+ oxidation state in their compounds [2]. Lanthanide ions (Ln^{3+}) demonstrate remarkable abilities to coordinate with oxygen and nitrogen atoms. Due to their elevated coordination number (ranging from 8 to 12 in solution) [3]. Among such ligands, azo compounds containing –N=N– groups are particularly attractive due to their strong chromophore properties, tautomerism, and excellent chelating ability One or more [4, 6]. Azo ligands, recognized for their remarkable chelating properties with various metal ions, also show significant biological activity due to their O and N donor atoms [7]. As the major ligand, we have selected 1-(2-pyridylazo)-2-naphthol, usually known as HPAN, with the letter H denoting the dissociable phenolic proton. Operating as a tridentate N,N,O-donor and generating two adjacent five-membered chelate rings, this ligand is known for its capacity to bind to metal centers via the dissociation of the acidic proton [8]. Although the coordination chemistry of PAN with various transition metals has been reported, studies on lanthanum (III) complexes remain scarce, and investigations of mixed-ligand systems involving PAN with biologically active ligands such as 1,10-phenanthroline (Phen) and 8-hydroxyquinoline (8HQ) are limited. Furthermore, little is known about how such complexes influence antibacterial activity. In this study, we report the synthesis and characterization of lanthanum (III) complexes with PAN as the primary ligand and Phen or 8HQ as secondary ligands. The complexes were characterized by spectroscopic and analytical methods, and their antibacterial activity was evaluated. This work addresses the gap in understanding the coordination behavior and biological potential of La(III) mixed-ligand complexes containing azo ligands.

Experimental

1-Materials and Instruments

All compounds utilized in this study, were reagent grad (E.Merk and BDH) including 1-(-2-pyridyl-azo)-2-naphthol (PAN), 8-hydroxyquinoline, 1,10-phenanthroline, $La(NO_3)_3 \cdot 6H_2O$, absolute ethanol, NaOH, and diethyl ether, Distilled water was utilized throughout the experiment. The Electrothermal 9100 apparatus was utilized to determine melting points in the UK. FT-IR spectra were recorded employing a Shimadzu spectrophotometer with KBr discs over the range of 4000 to 400 cm^{-1}. Carbon, hydrogen, and nitrogen (C, H, N) analysis was conducted using a Euro EA 3000 elemental analyzer from Italy. The ^{1}H-NMR spectra were performed on Bruker Analytic, 300 MHz. Electronic spectra were recorded in DMF solvent with (10^{-4}M) solution using (AEUV1609 (UK) CO.), LTD, UV-Vis-spectrometer with 1 cm quartz cell within 200-800 nm range. A digital Phillips pH-meter model pw9420 with a combined glass electrode was used to record the pH values.

2. Synthesis of Complexes

2.1. Synthesis of $[La(PAN)_2(NO_3)]$

A 1mmol solution of PAN (0.249 g) in 20 mL of ethanol was combined with 0.5mmol of $La(NO_3)_3 \cdot 6H_2O$ (0.2165 g) diluted in 10 mL of ethanol. The pH was modified to 8.6 by the addition of 0.1 M NaOH while stirring for 13 hours, resulting in a dark red precipitate. The mixture was cooled to 3–4 °C and held for 24 hours. The precipitate was further filtered and refined using multiple ether extractions yielding %83.

$$La(NO_3)_3.6H_2O + 2\,PAN \xrightarrow[PH=8\text{-}10]{EtOH} [La(PAN)_2(NO_3)]$$

Metal salt — Ligand — Bis-(pyridylazo)naphthol Lanthanium(III) nitrate

2.2. Synthesis of $[Ln(PAN)_2 (L_2)]$ when L_2= Phen, 8HQ.

To synthesize the complexes, 1mmol (0.249 g) of PAN (L_1) was combined with 0.5mmol of a secondary ligand L_2 either 8HQ (0.0725 g), or Phen (0.099 g) in ethanol. 0.5mmol (0.2165 g) of lanthanum nitrate (III) was dissolved in ethanol. The pH was modified to (8–10) with 0.1 M NaOH. Each ligand L_2 has unique conditions: for Phen, agitate the reaction mixture for 10 hours., then cool it in an ice bath for two hours; for 8HQ, stir the reaction mixture for 10 hours, resulting in the formation of a dark purple precipitate. The solutions were refrigerated to 3-4 °C and maintained at that temperature for 24 hours. The precipitate was subjected to filtration and refined through successive ether extraction. yield of 8HQ and Phen are %89and %81 respectively.

$$La(NO_3)_3.6H_2O + 2\,PAN + Phen \xrightarrow[PH=9.5]{EtOH} [La(PAN)_2(Phen)(NO_3)]$$

$$La(NO_3)_3.6H_2O + 2\,PAN + 8HQ \xrightarrow[PH=10]{EtOH} [La(PAN)_2(8HQ)]$$

3. Results and Discussion

3.1. Analytical and Physicochemical Characterization of the Complexes

The analytical results, together with the physical properties and molar conductivity of the prepared complexes, are presented in **Table 1.** The experimentally determined elemental composition closely matches the theoretical values, with deviations typically ranging between ±0.5. These minor discrepancies can be attributed to instrumental limitations, the hygroscopic nature of the samples, or partial retention of solvent molecules after drying. The finding supports the suggested molecular formulations of the complexes. All synthesized compounds exhibit non-electrolytic behavior, as indication by their low molar conductivity values (10^{-3} M in DMF at 32 °C). They are obtained as stable, colored powders with high melting points, insoluble in water but readily soluble in DMF and DMSO.

Table.1: Some physical properties, theoretical and experimental analytical results of complexes

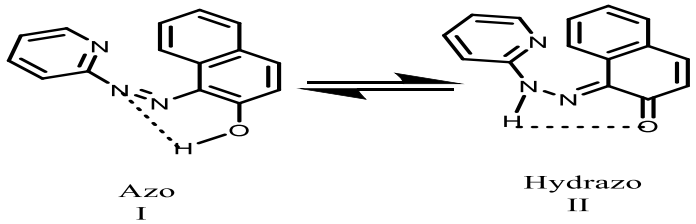
Formula of the Complexes	Melting Point (^{0}C)	Color	Molecular weight	(Calculated) Found%			Molar conductivity (cm^2. ohm^{-1}. mol^{-1})
				C	H	N	
PAN $C_{15}H_{11}N_3O$	142	Orange	249.27	(72.3) 72.01	(4.4) 4.2	(16.9) 16.3	----
La: 2PAN $LaC_{30}H_{20}N_7O_5$	$d.p$ >300	Red	697.43	(51.66) 52.1	(2.89) 2.01	(14.06) 13.52	11.82
La: 2PAN: Phen $LaC_{42}H_{28}N_9O_5$	$d.p$ 261	Intense brown	877.64	(57.46) 56.79	(3.22) 3.12	(14.36) 13.91	14.64
La: 2PAN: 8HQ $LaC_{39}H_{26}N_7O_3$	$d.p$ 252	Purple	779.59	(60.09) 59.03	(3.36) 2.53	(12.58) 12.06	9.33

-Values in brackets = theoretical (calculated)
-Values without brackets = experimental (practical, found)

3.2. Spectroscopic characterization

3.2.1. IR spectroscopy for PAN and $[La(PAN)_2(NO_3)]$ complex.

The chemical grafting of 1-(2-pyridylazo)-2-naphthol (PAN) was validated by FT-IR spectroscopy within the 400–4000 cm^{-1} range utilizing KBr. The spectra of free PAN exhibited distinct peaks at (3047, 1622, 1606, 1402) cm^{-1}, aligning with conventional PAN. The lack or diminishment of the (3200–3500 cm^{-1}) band signifies robust intramolecular hydrogen bonding between the naphthol –OH group and the azo nitrogen [9]. As shown in (I and II), the ligand can be in a state of equilibrium between its azo and hydrazone forms. The intense absorption band seen in the ligand's spectrum at 1606 cm^{-1} probably relates to the C=O stretching of the hydrogen-bonded ring structures seen in the hydrazone form. While carbonyl stretching bands are generally seen around 1700 cm^{-1}, a red shift is seen for hydrogen-bonded ring systems. Due to the involvement of the C=O bond, the absorption band at 1606 cm^{-1} moves to a lower wavenumber, 1593 cm^{-1} in the spectra of the metal complexes [10].



Azo
I

Hydrazo
II

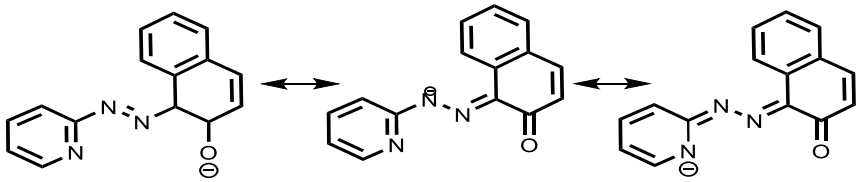
Within the molecule, the C=N bond goes from (1622 to 1614 cm^{-1}), therefore lowering its wave number [11]. Between 989 and 952 cm^{-1}, the infrared spectrum of the ligand shows a weak band related to the υ(C–N)py, when the ligand complexes, this band is seen to shift to a lower frequency range of (896 to 860 cm^{-1}), a change caused by coordination including the nitrogen atom of the pyridine ring [10]. The most prominent characteristic in the PAN spectrum is the N=N vibrational stretch, which shows at 1402 cm^{-1}. Upon complex production, this band shifts to a lower frequency of 25 cm^{-1}, therefore positioning it at 1377 cm^{-1}. This points to this group's interaction with the metal ion [12]. The stretching vibrations ν(N-O) are found in the range (1280–1475 cm^{-1}). Splitting observed in this range is in favor of bidentate coordination of the nitrate groups [13].The spectral bands noted at (505–598cm^{-1}) and (635–669cm^{-1}) represent La–N and La–O vibrational modes correspondingly, therefore indicating the beginning of metal–ligand interactions.

3.2.2. IR spectroscopy for mixed ligand complexes

In the free HQ ligand, the C=N mode is observed at 1579 cm^{-1}, while in the complex spectra it is shifted to lower wavenumbers, around from (1544 to 1496 cm^{-1}) [14].The lack of the band around 3440 cm^{-1}, due to the O–H stretching of the free 8HQ, suggests that complexation is through deprotonation of the hydroxyl group of the 8HQ moiety [15]. The shifts observed for the C=N and N=N stretching bands are consistent with those discussed in Section **(3.2.1)**, further confirming coordination of PAN through both the azomethine and azo groups. In **figure (1)**. New, weak bands at around (653-597 cm^{-1}) and 503 cm^{-1} are assigned to M–O and M–N vibrations, respectively. These vibration bands are absent in the infrared spectra of 8HQ and PAN. The C=N stretching vibration of the free 1,10-phenanthroline ligands, which is at 1647 cm^{-1}, is shifted to a slightly lower value of 1560 cm^{-1} due to bonding through the amino group, as in similar structures [16]. The coordination centers were also established by the presence of υ(M–N) and υ(M–O) stretching bands, which were seen approximately in the ranges (418–437 cm^{-1}) and (588–634 cm^{-1}), respectively. The N–O stretching bands are found in the region (1315–1463 cm^{-1}), and the splitting observed shows that the nitrate groups are present as bidentate ligands [17].

3.3. ^{1}H-NMR spectroscopy

In this paper, we investigated the solution structure of 1-(2-pyridylazo)-2-naphthol (PAN) using ^{13}C and ^{1}H NMR in DMSO show in **Figure (2)**. The evidence indicates that PAN exists predominantly as the quinone–hydrazone tautomer, a finding corroborated by the carbonyl carbon resonances observed in the ^{13}C-NMR spectrum at 180 ppm [18]. ^{1}H NMR spectra of PAN, which exhibits a singlet at 15.98 ppm due to the azo OH proton. All the other proton signals are in the range of (6–8) ppm [11]. Under basic conditions, HPAN is deprotonated, producing the PAN anion [19]. Upon complexation, this signal disappears, due to coordination through the oxygen atom. Additionally, the aromatic and heteroaromatic protons undergo downfield shifts, indicating perturbation of the electronic environment due coordination through both the azo and pyridyl nitrogen. These spectral changes are consistent with the proposed tridentate binding mode of PAN. **Figure (3)** shows the ^{1}H NMR spectra $[La(PAN)_2(NO_3)]$ complex.



III

The chemical shifts of the protons in the ^{1}H-NMR spectra of the complexes of $[La(PAN)_2(NO_3)]$,8HQ, and Phen, are reported in **Table (2)** and **figure (4)** showed ^{1}H-NMR spectra of $[La(PAN)_2(Phen)(NO_3)]$ complex.

Table .2: ^{1}H-NMR chemical shifts for the prepared complexes.

Complexes	(ppm), multiplicity, intensity, and assignment
$[La(PAN)_2(NO_3)]$	^{1}H NMR (300 MHz, DMSO-d_6) δ 8.58 (d, J = 5.1 Hz, 2H), 8.45 – 8.42 (m, 2H), 8.02 – 7.98 (m, 4H), 7.92 (dd, J = 7.9 Hz, 2H), 7.81 – 7.71 (m, 4H), 7.50 (ddd, J = 7.5, 1.3 Hz, 2H), 7.39 (dd, J = 8.4 Hz, 2H), 6.71 (d, J = 9.7 Hz, 2H).
$[La(PAN)_2(8HQ)]$	^{1}H NMR (500 MHz, DMSO-d^6) δ 8.11 (dd, J = 7.1, 2.0 Hz, 2H), 8.00 (d, J = 7.4, 1.6 Hz, 1H), 7.90 – 7.79 (m, 4H), 7.68 (t, J = 7.5 Hz, 1H), 7.51 – 7.42 (m, 3H), 7.41 – 7.31 (m, 2H), 7.26 (d, J = 7.5, 1.7 Hz, 1H), 6.77 (dd, J = 7.5, 1.7 Hz, 2H).
$[La(PAN)_2(Phen)(NO_3)]$	^{1}H NMR (300 MHz, DMSO-$d6$) δ 9.12 (dd, J = 4.3, 1.8 Hz, 1H), 8.52 (dd, J = 8.1, 1.8 Hz, 1H), 8.42 (t, 1H), 8.01 (d, J = 7.5 Hz, 1H), 7.80 (dd, 1H), 7.72 (d, J = 7.7 Hz, 1H), 7.61 (s, 1H), 7.51 (d, J = 7.4 Hz, 1H), 7.29 (dt, J = 5.5, 2.7 Hz, 1H), 7.08 (s, 1H), 6.71 (d, J = 9.7 Hz, 1H), 6.48 (d, 1H).

3.4. UV-spectra of ligand and complexes

The ultraviolet-visible (UV-vis) spectra of all the complexes were studied in dimethylformamide (DMF) at a concentration of 1×10^{-4} M. As shown in **Figure (5)**, the UV-vis spectrum of the PAN ligand, as well as that of the $[La(PAN)_2(NO_3)]$ complex, displays three main absorption bands: namely, at 226 nm and 305 nm, attributed to π→π* transitions in the pyridine and naphthol rings, respectively [20]. and a strong band at 466 nm due to charge transfer from the electron-rich naphthol ring to the pyridine ring through the azo (–N=N–) bridge, which is responsible for the ligand's orange-red color. The red shift of the 466 nm band upon complexation signifies the coordination of La^{3+} with the azo nitrogen. A shoulder shift to shorter wavelengths indicates metal binding to the deprotonated hydroxyl oxygen [21]. A shoulder at about 410 nm is associated with the principal peak at 466 nm, likely because of unsaturated functions such as C=O or C=N, supporting the existence of the Quino

hydrazo form of the PAN ligand [22]. The complexes formed by the ligand mixture of Phen and 8HQ exhibit a red shift relative to the La (PAN) complex, accompanied by a notable increase in absorption intensity. and **Table (3)** presents the spectra obtained for these complexes alongside the spectra of the ligands for comparative analysis.

Table .3: UV-vis spectrum results of lanthanum (III) complexes

Compounds	(λ max) (nm)	Complexes	(λ max) (nm)
PAN	466,410,305,226	**[La(PAN)$_2$(NO$_3$)]**	518,390,314,271
Phen	264,230	**[La(PAN)$_2$(Phen)(NO$_3$)]**	472,306,292
8HQ	318,242,203	**[La(PAN)$_2$(8HQ)]**	488,306,285

3.5. Biological activity

The in vitro antibacterial potential of the synthesized lanthanum complexes was assessed using the agar disc diffusion technique against two clinically relevant bacterial strains: Staphylococcus aureus (Gram-positive) and Escherichia coli (Gram-negative). The assays were performed at four concentration levels (200, 400, 600, and 800 ppm), with dimethylformamide (DMF) employed as the solvent. Following inoculation, the agar plates were incubated at 37 °C for 24 hours. Antibacterial efficacy was determined by recording the diameter of the inhibition zones (mm), as summarized in **Table (4)** and illustrated in **Figure (6)**.

Table .4: Results obtained from the biological activity of the prepared complexes

Compound	**G (+) S. aureus (200, 400, 600, 800ppm)**				**E. coli G (-), (200, 400, 600, 800 ppm)**			
[La(PAN)$_2$ (NO$_3$)]	20mm	26mm	25mm	29mm	23mm	21mm	22mm	21mm
[La(PAN)$_2$(Phen)(NO$_3$)]	22mm	19mm	23mm	20mm	21mm	20mm	22mm	20mm
[La(PAN)$_2$(8HQ)]	27mm	26mm	28mm	30mm	24mm	25mm	19mm	23mm
CEF	20mm				0			
DMF	0				0			

Inhibition zone of Staphylococcus aureus
susceptible ≥21mm, intermidate14-20mm, Resistance ≤13mm

4. conclusions

The lanthanum (III) complexes obtained in this study exhibit stable coordination environments, with PAN acting as a tridentate ligand through the azo nitrogen, pyridyl nitrogen, and phenolic oxygen atoms. In the $[La(PAN)_2(NO_3)]$ complex, the nitrate ion coordinates in a bidentate manner, leading to an overall coordination number of eight. In the mixed-ligand systems, additional donor sites from 1,10-phenanthroline or 8-hydroxyquinoline expand the coordination sphere, resulting in octa- to Deca-coordinated geometries that are typical for lanthanides. Spectroscopic evidence including IR shifts of C=N and N=N groups, disappearance of the O–H band in 8HQ, downfield ^{1}H-NMR signals, and UV–Vis charge-transfer transitions—supports the involvement of azo, pyridyl, and hydroxyl donor atoms in binding. These results confirm the effective formation of mixed-ligand La(III) complexes and provide new structural insight into the coordination versatility of lanthanides with azo- and N,O-donor ligands.

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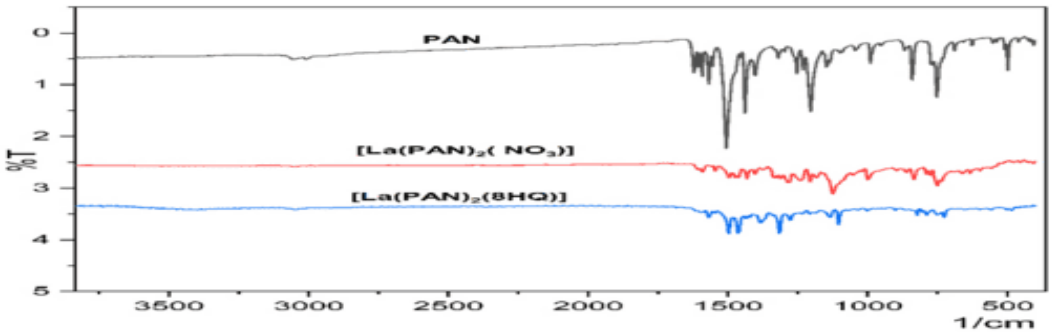


Fig.1: FT-IR spectrum of PAN, $[La(PAN)_2(NO_3)]$ and $[La(PAN)_2(8HQ)]$ complex

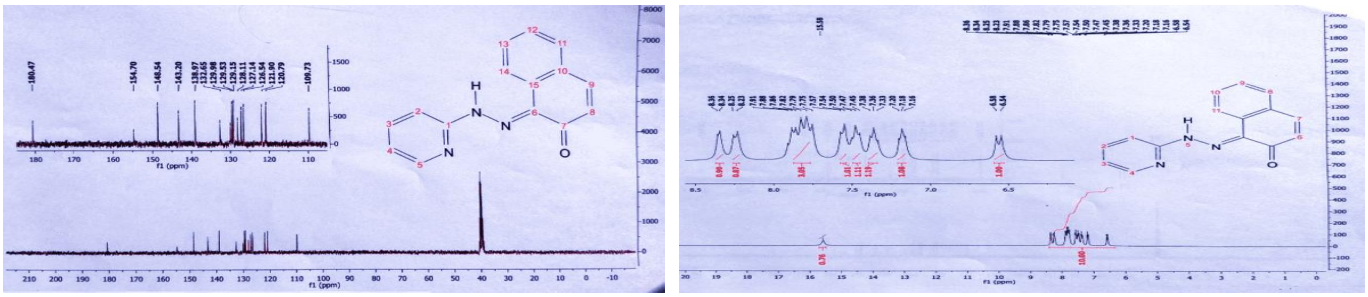


Fig .2: ^{13}C and ^{1}H-NMR spectrum of PAN

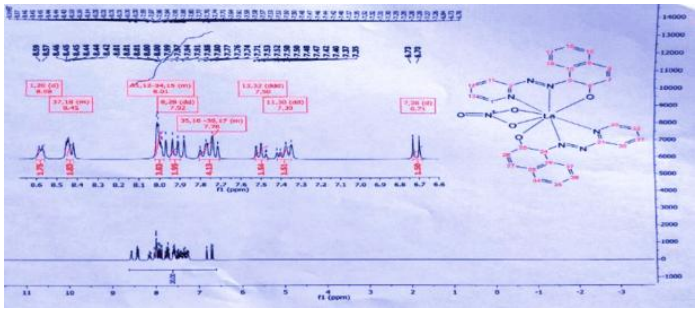


Fig .3: ^{1}H-NMR spectrum of PAN and $[La(PAN)_2(NO_3)]$

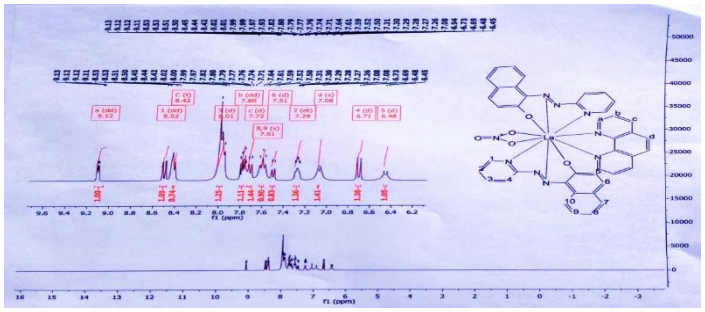


Fig .4: ^{1}H-NMR spectrum of $[La(PAN)_2(Phen)(NO_3)]$ complex.

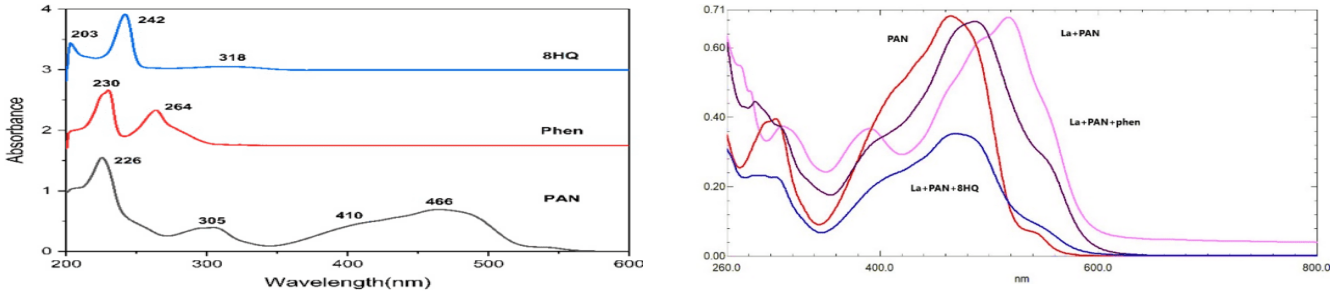
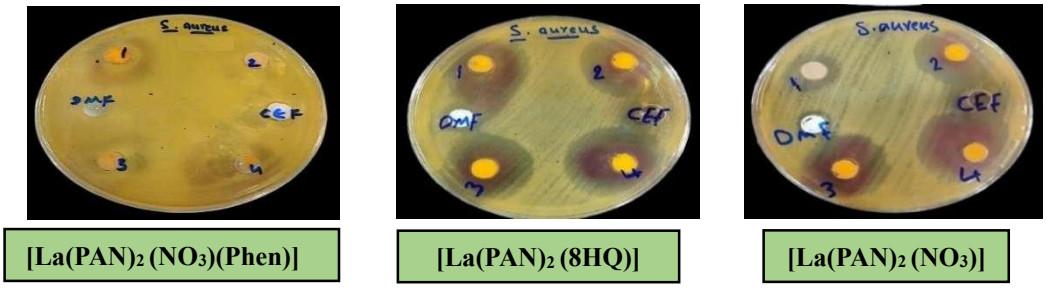


Figure.5: UV-vis spectra of all ligands and their complexes



[La(PAN)$_2$ (NO$_3$)(Phen)] [La(PAN)$_2$ (8HQ)] [La(PAN)$_2$ (NO$_3$)]

Figure.6: biological activity of prepared complexes