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# NOVEL MIXED-LIGAND NEODYMIUM(III) COMPLEXES: SYNTHESIS, CHARACTERIZATION, AND BIOLOGICAL INSIGHTS

Chemistry

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## ABSTRACT

The present study comprise synthesis, spectroscopic studies and biological evaluation of new mixed ligand Neodymium complexes. These Nd(III) complexes having composition $[M(1N2N)_2 \cdot (L) \cdot 2H_2O]$ were synthesized by the reaction of $NdCl_3 \cdot 6H_2O$ salt with 1-nitroso-2-naphthol (1N2N) as the primary ligand and different amino acids like L-alanine, L-hydroxyproline, L-tryptophan, and L-tyrosine as secondary ligands. The synthezied complexes were characterized by Molar conductivity, TGA, UV-VIS and FT-IR Spectroscopic techniques. The conductance analysis revealed the non-electrolytic nature of complexes. The TGA study showed good concordance with complicated structure of Complex. The coordination of primary and secondary ligand with neodymium metal ion in the complexes was confirmed by the FT-IR spectra. Furthermore, the biological study was done by employing the agar cup method to screen against selected bacteria—*E. coli, B. Cereus, S. aureus and K. Pneumonia*. These study showed the antibacterial nature of the synthesized mixed ligand Neodymium complexes.

## KEYWORDS

Mixed ligand complexes, Neodymium, amino acid, Antibacterial activity, 1-nitroso-2-naphthol

## INTRODUCTION

The chemical compound 1-Nitroso-2-naphthol is a versatile one. This well-known gravimetric reagent is used to measure metal ions spectrophotometrically[1]. It creates complexes with transition, inner transition and alkali metals successively. It has been observed that metal complexes containing 8-hydroxyquinoline and 1-nitroso-2-naphthol have antifungal and antibacterial properties[2]. Strong antibacterial action is inhibited by the lanthanum and cerium complexes with amino acids and 1-nitroso-2-naphthol[3]. It is widely known that schiff-base camphor and 4-aminobenzoic acid can be used to synthesise Praseodymium and Neodymium complexes[4]. Neodymium complexes exhibit noteworthy antibacterial and anticancer properties[5-7]. There have been reports on the synthesis and characterisation of rare earth metal complexes with amino acids[8-9]. From our extensive literature search, it was observed that, the mixed ligand complexes of Nd(III) with 1-nitroso-2-naphthol (1N2N) as the primary ligand and amino acid as secondary ligand was not reported yet. Considering the biological importance of mixed ligand complexes having Neodymium metal as well as 1-Nitroso-2-naphthol and amino acid ligand, it is higly desirable to synthesize Nd(III) complex with 1-nitroso-2-naphthol (1N2N) as the primary ligand and amino acid as secondary ligand. In this context, we envisioned the synthesis of mixed ligand Nd(III) complex with 1-nitroso-2-naphthol (1N2N) as the primary ligand and amino acid as secondary ligand. The present study includes the synthesis, structural analysis, and antibacterial activity of some new mixed ligand Neodymium (III) complexes with 1-nitroso-2-naphthol and L-alanine, L-tryptophan, L-hydroxyproline, and L-tyrosine acting as a primary and secondary ligands.

### Experimental

In laboratory work, analytical grade Neodymium chloride hexahydrate metal salt, 1-nitroso-2-naphthol, L-alanine, L-hydroxyproline, L-tryptophan, L-tyrosine ligands and Dimethyl sulfoxide (DMSO), ethanol, Dimethyl Formamide (DMF) solvents were utilized as such as purchased from Loba chemicals.

### Instrumentation

Using the EA300A 67 Elemental Analyzer, carbon, nitrogen, and hydrogen have all undergone microanalysis. On a Shimadzu UV-Visible spectrophotometer 1800, the electronic spectrum of a solution of synthesised compounds in DMF ($10^{-3}$ M) was recorded. Using an EQ-667 digital conductometer, the molar conductance of a solution of synthesised complexes in DMF was measured. The magnetic susceptibility of the complexes prepared in this study was measured employing the Gouy balance technique, with Hg[Co(SCN)□] used as the reference standard for calibration. Using dry potassium bromide as a standard reference, the FT-IR spectra of synthesised Nd(III) complexes was captured using an FT-IR Shimadzu spectrophotometer.

### Preparation of Neodymium (III) Complexes

The reaction of $NdCl_3 \cdot 6H_2O$ with primary ligand 1-nitroso-2-naphthol and selected secondary ligand like L-alanine, L-hydroxyproline, L-tryptophan, and L-tyrosine was carried out to afford desired mixed ligand Neodymium (III) complexes.

The solutions of 0.359 g of Neodymium chloride hexahydrate (1 mmol) in 10 $cm^3$ distilled water, 0.346 g of 1-nitroso-2-naphthol (2 mmol) in 10 $cm^3$ ethyl alcohol and 1 mmol amino acids (L-alanine, L-hydroxyproline, L-tryptophan, L-tyrosine) in 10 $cm^3$ distilled water was prepared in separate glass beakers. The aqueous Neodymium chloride hexahydrate solution was gradually added to the ethanolic 1-nitroso-2-naphthol solution. This mixture was then digested for ten minutes. To this reaction mixture, the addition of aqueous amino acid solution was done. The reaction mixture was again heated in a hot water bath for ten minutes at 60 °C. After cooling the reaction mixture to room temperature, the diluted ammonia solution was gradually added until the formation of neutral precipitate complete. The reaction mixture was filtered to get crude residue. The obtained residue was then washed with alcohol followed by water. The product was then dried at 110 °C in an oven and used for Spectroscopic studies and Biological Evaluation.

### Antibacterial Study

The antibacterial property of synthesised Neodymium complexes were examined against *Klebsiella pneumoniae, Escherichia coli, Bacillus cereus and Staphylococcus aureus* selected bacteria[10].

This procedure involved filling a sterile petriplate with sterile nutrient agar that contained a bacterial strain. The petriplate was cooled until it solidified. Next, using a sterilized cork borer, wells with an interior diameter of 5 mm were created in each petriplate. The solution of Neodymium complexes in DMF (1mg/$cm^3$) were then added to the wells. The antibacterial property of complexes were determined by measuring the inhibitary zones after incubation of sterile plates for 24 hours in refrigerator at 37 °C. Lastly, the activity of the metal complexes was compared with standard drug tetracycline.

## RESULTS AND DISCUSSION:

### 1. Synthesis Reaction

The Neodymium complex formation reaction is shown below,

$$MCl_3.6H_2O + 2(1N2N) + HL \rightarrow [M(1N2N)_2 \cdot (L) \cdot 2H_2O] + 3HCl + 4H_2O$$

where M is the Neodymium chloride hexhydrate and 1N2N is 1-nitroso-2-naphthol, HL = L-alanine, L-hydroxyproline, L-tryptophan, and L-tyrosine.

### 2. Physico-Chemical Properties

The synthesized Nd(III) mixed ligand complexes were radish brown powder form and thermally stable. They were less soluble in DMSO and DMF and unsolvable in water and non-polar solvents. The physical properties of synthesized Neodymium complexes are presented in Table 1. Elemental analysis confirms a 1:2:1 stoichiometry for the synthesized complexes, in agreement with the proposed general formula $[Nd(1N2N)_2(L).2H_2O]$ (Table-2).

**Table 1: Physical Properties of Synthesized Neodymium Complexes**

| Complex | D.T./ M.P °C | M. Formula | M. Wt. (g) | Color |
|---|---|---|---|---|
| $[Nd(1N2N)_2(Ala)\cdot 2H_2O]$ | 220 | $C_{23}H_{22}N_3NdO_8$ | 612.68 | Reddish brown |
| $[Nd(1N2N)_2(Hpro)\cdot 2H_2O]$ | 240 | $C_{25}H_{24}N_3NdO_9$ | 654.71 | Reddish brown |
| $[Nd(1N2N)_2(Try)\cdot 2H_2O]$ | 215 | $C_{31}H_{27}N_4NdO_8$ | 727.81 | Reddish brown |
| $[Nd(1N2N)_2(Tyr)\cdot 2H_2O]$ | 205 | $C_{29}H_{26}N_3NdO_9$ | 704.77 | Reddish brown |

1N2N = 1-nitroso-2-naphthol, Ala = alanine , Hpro = hydroxyproline , Try = tryptophan , Tyr = tyrosine

**Table 2: Elemental Composition Analysis Data of Synthesized Neodymium Complexes**

| Complex | Elemental Analysis (%) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Nd | | C | | N | | H | |
| | Obs. | Cal. | Obs. | Cal. | Obs. | Cal. | Obs. | Cal. |
| $[Nd(1N2N)_2(Ala)\cdot 2H_2O]$ | 23.50 | 23.54 | 45.1 | 45.09 | 6.80 | 6.86 | 3.60 | 3.62 |
| $[Nd(1N2N)_2(Hpro)\cdot 2H_2O]$ | 22.10 | 22.03 | 45.80 | 45.86 | 6.38 | 6.42 | 3.65 | 3.69 |
| $[Nd(1N2N)_2(Try)\cdot 2H_2O]$ | 19.45 | 19.82 | 50.79 | 51.16 | 7.65 | 7.70 | 3.69 | 3.74 |
| $[Nd(1N2N)_2(Tyr)\cdot 2H_2O]$ | 20.41 | 20.47 | 49.40 | 49.42 | 5.93 | 5.96 | 3.70 | 3.72 |

**Molar Conductivity and Magnetic Susceptibilty Measurements**

The electrical conductivity of Neodymium complexes having concentration 0.001M in DMF was measured. The conductance values were found in the range of $0.129x10^{-3}$ to $0.193x10^{-3}$ Mhos $cm^2$ $mol^{-1}$. The small conductance value indicate that complexes were not electrolytic in nature (11). The magnetic moments of neodymium complexes were determined from their experimentally measured magnetic susceptibilities after applying the appropriate diamagnetic corrections revealed their paramagnetic behaviour (12-13). The obtained magnetic moment and molar conductivity data of complxes are listed in Table-3.

**Table 3: Molar Conductivity and Magnetic Susceptibilty Data of Nd (III) Complexes.**

| Sr. No. | Complex | Λm (Mhos $cm^2$ $mol^{-1}$) | Xm | Xg | μeff (B.M.) |
|---|---|---|---|---|---|
| 1 | $[Nd(1N2N)_2(Ala)\cdot 2H_2O]$ | $0.129 \times 10^{-3}$ | $3.89781 \times 10^{-3}$ | $6.35 \times 10^{-6}$ | 3.09 |
| 2 | $[Nd(1N2N)_2(Hpro)\cdot 2H_2O]$ | $0.176 \times 10^{-3}$ | $4.05920 \times 10^{-3}$ | $6.20 \times 10^{-6}$ | 3.17 |
| 3 | $[Nd(1N2N)_2(Try)\cdot 2H_2O]$ | $0.184 \times 10^{-3}$ | $4.20674 \times 10^{-3}$ | $5.78 \times 10^{-6}$ | 3.23 |
| 4 | $[Nd(1N2N)_2(Tyr)\cdot 2H_2O]$ | $0.193 \times 10^{-3}$ | $3.9326 \times 10^{-3}$ | $5.58 \times 10^{-6}$ | 3.12 |

**Infra-red Spectra**

The FT-IR spectral analytical data of synthesized Neodymium complexes are shown in Table 5. The data displays the peak for hydroxyl group of water molecule at 3203–3381 $cm^{-1}$. The -OH group of the 1-nitroso-2-naphthol underwent deprotonation during complexation, as indicated by the nonappearance of signal at about 3448 $cm^{-1}$. Moreover, 1155 $cm^{-1}$ peak suggested that, the complex included a 1-nitroso-2-naphthol moiety. Additionally, the 1-nitroso-2-naphthol is bonded with Neodymium metal ions through nitrogen atom, as indicated by the peak in the range of 1230-1211 $cm^{-1}$ and 1577-1552 $cm^{-1}$ for nitrile and nitroso groups respectively. Furthermore, the amino group coordination was verified by the bands in the 3132-3113 $cm^{-1}$ and 3061-3039 $cm^{-1}$ ranges, in addition to the nitrile peak region at 840-839 $cm^{-1}$. The peak found at 1616-1614 $cm^{-1}$ and 1400-1392 $cm^{-1}$ was used to identify the presence of the -COOH group of amino acid molecule. Ultimately, weak bands in the 526–509 $cm^{-1}$ region and at 638–636 $cm^{-1}$ region confirmed the metal–nitrogen and metal–oxygen interaction.

**Table 4: FT-IR bands ($cm^{-1}$) of Synthesized Neodymium Complexes**

| Sr. No. | Complex | FT-IR Peaks ($cm^{-1}$) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | ν O-H $H_2O$ | ν C-O Sym. A.a | ν N-H Sym. A.a. | ν C=O Asym. A.a | ν N-H Asym. A.a | ν C-N A.a | ν C-O (1N2N) | ν N=O (1N2N) | ν C-N (1N2N) | ν M-N | ν M-O |
| 1 | $[Nd(1N2N)_2(Ala)\cdot 2H_2O]$ | 3361(b) | 1396(m) | 3061 (w) | 1616(s) | 3122(w) | 839(m) | 1155(w) | 1552(s) | 1211(m) | 509(w) | 636(w) |
| 2 | $[Nd(1N2N)_2(Hpro)\cdot 2H_2O]$ | 3381(b) | 1392(w) | 3061(w) | 1616(s) | 3132(w) | 839(m) | 1155(w) | 1577(s) | 1230(m) | 522(w) | 636(w) |
| 3 | $[Nd(1N2N)_2(Try)\cdot 2H_2O]$ | 3346(b) | 1398(w) | 3059(w) | 1616(s) | 3115(w) | 839(m) | 1155(w) | 1554(s) | 1213(m) | 522(w) | 636(w) |
| 4 | $[Nd(1N2N)_2(Tyr)\cdot 2H_2O]$ | 3203(b) | 1400(w) | 3039(w) | 1614(s) | 3113(w) | 840(m) | 1155(w) | 1556(s) | 1213(m) | 526(w) | 638(w) |

1N2N = 1-nitroso-2-naphthol, b = broad band, m = medium band, w = weak band, s = strong band, A.a = Amino Acids, Sym. = Symmetric, Asym. = Asymmetric

**Electronic Absorption Spectra**

UV-visible (200-800 nm) spectrums of all synthesised Neodymium complexes with 0.001 molar concentration in dimethyl formamide were recorded. The Nd (III) complexes revealed three peaks in their Uv-Vis spectrum. The charge transfer L→M, $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ transitions were verified by the bands spanning 436-440 nm, 263-269 nm, and 380-389 nm, respectively (14). The collected Uv-Vis spectral data is summarised in Table-5.

**Table 5: Electronic Absorption Spectral Data of Synthesized Neodymium Complexes**.

| Sr. No. | Complex | ν ($cm^{-1}$) | λ (nm) | Assignments |
|---|---|---|---|---|
| 1 | $[Nd(1N2N)_2(Ala)\cdot 2H_2O]$ | 22727 | 440 | Charge-transfer |
| | | 26315 | 380 | $n \rightarrow \pi^*$ |
| | | 37735 | 265 | $\pi \rightarrow \pi^*$ |
| 2 | $[Nd(1N2N)_2(Hpro)\cdot 2H_2O]$ | 22935 | 436 | Charge-transfer |
| | | 25706 | 389 | $n \rightarrow \pi^*$ |
| | | 37174 | 269 | $\pi \rightarrow \pi^*$ |
| 3 | $[Nd(1N2N)_2(Try)\cdot 2H_2O]$ | 22779 | 439 | Charge-transfer |
| | | 26109 | 383 | $n \rightarrow \pi^*$ |
| | | 37313 | 268 | $\pi \rightarrow \pi^*$ |
| 4 | $[Nd(1N2N)_2(Tyr)\cdot 2H_2O]$ | 22935 | 436 | Charge-transfer |
| | | 25773 | 388 | $n \rightarrow \pi^*$ |
| | | 38022 | 263 | $\pi \rightarrow \pi^*$ |

**Thermogravimetric Analysis**

The Synthesized Neodymium complexes were subjected to a steady heating rate of 10 °C/min in an inert nitrogen environment for the thermogravimetric examination. The complexes were studied for TGA investigations to know degradation stages and its temperature. The obtained TGA data are summarized in Table-6. The TGA study showed three distict decomposition stages. The complex first decomposes quickly, losing 5.3 - 6.0 % of its weight in the temperature range 105 - 160 °C as a result of elimination of two water molecules. The second breakdown step showed 15.33 - 27.03 weight loss between 190 - 280 °C temperature range due to elimination of amino acid moiety from the complexes. The final step showed 44.0 – 54.0 % weight loss due to breakdown of 1-nitroso-2-naphthol in the temperature range 190-280 °C. After 648 °C, a consistent weight plateau appears, signifying that the complexes converted into $Nd_2O_3$ fine powder. This was further confirmed by XRD analysis of $Nd_2O_3$ product[15].

**Table 6: Thermal Analysis Results For Neodymium Complexes**

| Sr. No. | Complex | % Loss of Weight | | Residue | Temp. Range (°C) |
|---|---|---|---|---|---|
| | | Calculated | Found | | |
| 1 | $[Nd(1N2N)_2(Ala)\cdot 2H_2O]$ | 5.87 | 6.00 | $[Nd(1N2N)_2(Ala)]$ | 105-145 |
| | | 14.54 | 15.33 | $[Nd(1N2N)_2]$ | 200-260 |

| | | | | | |
|---|---|---|---|---|---|
| | | 56.52 | 54.00 | $[Nd_2O_3]$ | 280-680 |
| 2 | $[Nd(1N2N)_2(Hpro)\cdot 2H_2O]$ | 5.50 | 5.3 | $[Nd(1N2N)_2(Hpro)]$ | 120-160 |
| | | 20.03 | 20.07 | $[Nd(1N2N)_2]$ | 190-280 |
| | | 49.76 | 49.33 | $[Nd_2O_3]$ | 360-660 |
| 3 | $[Nd(1N2N)_2(Try)\cdot 2H_2O]$ | 4.86 | 5.3 | $[Nd(1N2N)_2(Try)]$ | 115-155 |
| | | 27.53 | 27.03 | $[Nd(1N2N)_2]$ | 190-270 |
| | | 43.92 | 44.00 | $[Nd_2O_3]$ | 310-650 |
| 4 | $[Nd(1N2N)_2(Tyr)\cdot 2H_2O]$ | 5.10 | 5.3 | $[Nd(1N2N)_2(Tyr)]$ | 115-155 |
| | | 25.63 | 24.07 | $[Nd(1N2N)_2]$ | 200-280 |
| | | 46.10 | 46.66 | $[Nd_2O_3]$ | 335-660 |

**Antibacterial Studies**

All synthesized new Neodymium mixed ligand complexes showed antibacterial property against selected pathogenic bacteria *S. aureus, B. cereus, E. coli and K. pneumoniae* respectively. Table -7 represents zone of inhibition of selected organisms in millimeters.

This approach showed that all Neodymium complexes exhibhit greater antibacterial activity against B. cereus and K. pneumoniae as compared to S. aureus and E. coli bacteria. The findings demonstrate that, in comparison to parent metal salts and ligands, Neodymium complexes have greater antibacterial activity. The chelate action leads to increased Neodymium complex activity. The chelation process reduces the metal ions observable polarity, makes the chelate more hydrophobic, and allows it to pass through the lipid layer of microorganisms. [16]

**Table 7: Biological Activity (mm) of Synthesized Neodymium Metal Complexes**

| Sr. No. | Neodymium Complexes | Zone of Inhibition in mm | | | |
|---|---|---|---|---|---|
| | | *K. pneumoniae* | *E. coli* | *B. cereus* | *S. Aureus* |
| 1 | $[Nd(1N2N)_2(Ala)\cdot 2H_2O]$ | 25 | 19 | 25 | 20 |
| 2 | $[Nd(1N2N)_2(Hpro)\cdot 2H_2O]$ | 24 | 17 | 25 | 18 |
| 3 | $[Nd(1N2N)_2(Try)\cdot 2H_2O]$ | 23 | 15 | 24 | 17 |
| 4 | $[Nd(1N2N)_2(Tyr)\cdot 2H_2O]$ | 24 | 18 | 26 | 19 |

On the basis of physicochemical results and spectral study data, the anticipated structure of Neodymium complexes can be represented as shown in Figure 1,2,3,4 respectively.

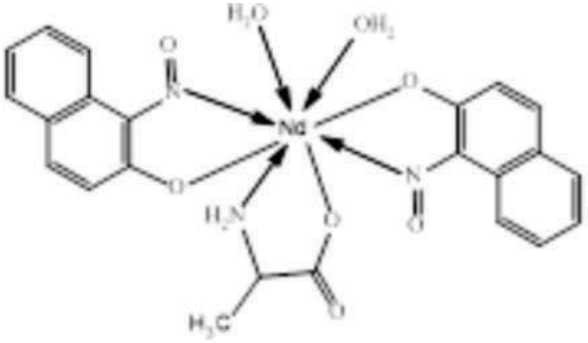

**Figure1:** Proposed structure of $[Nd(1N2N)_2(Ala)\cdot 2H_2O]$

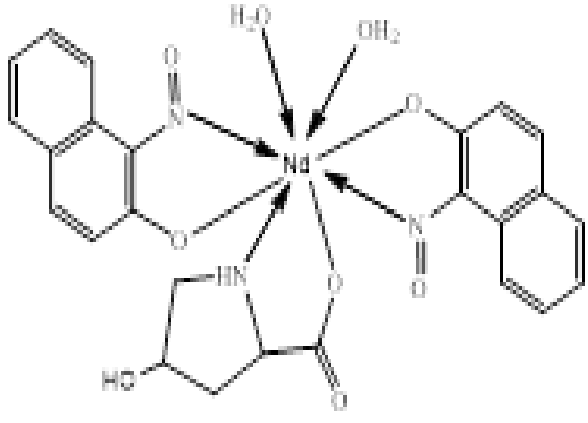

**Figure2:** Proposed structure of $[Nd(1N2N)_2(Hpro)\cdot 2H_2O]$

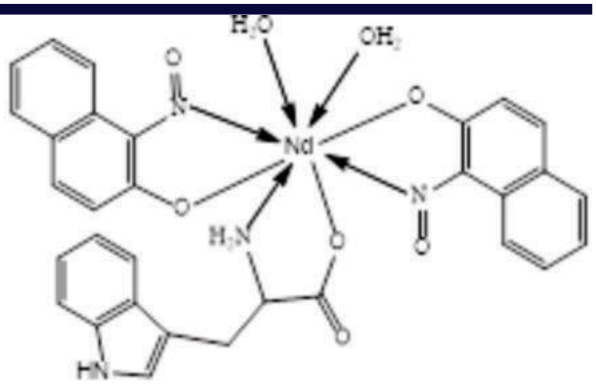

**Figure 3:** Proposed structure of $[Nd(1N2N)_2(Try)\cdot 2H_2O]$

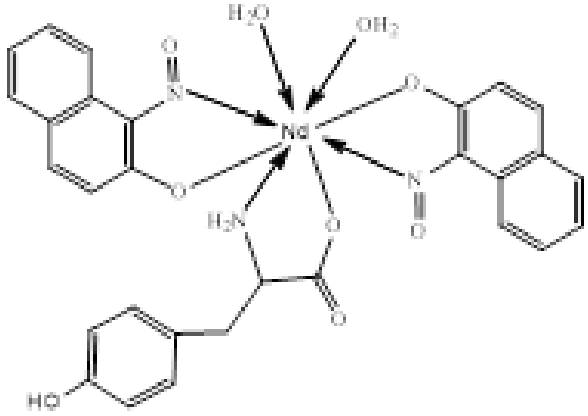

**Figure 4:** Proposed structure of $[Nd(1N2N)_2(Tyr)\cdot 2H_2O]$

## CONCLUSIONS

We have effectively accomplished new mixed ligand Neodymium complexes with 1-nitroso-2-naphthol and several amino acids (L-alanine, L-hydroxyproline, L-tryptophan, and L-tyrosine) as a secondary ligand. The stoichiometry of synthesized complex was revealed as 1:2:1 by elemental analysis. The molar conductance values less than one confirmed that synthesized Neodymium complexes were non electrolytes. In complexes, the coordination of Neodymium metal ion with water molecules was validated by the TGA data. The metal-ligand bonding through donor O- and N-atoms was verified by the FT-IR spectra. The charge transfer, $n \rightarrow \pi^*$, $\pi \rightarrow \pi^*$ transitions were revealed via the electronic absorption study. It was observed that the antibacterial activity of complexes is greater than that of their corresponding parent ligands. The aforementioned findings validate coordination number 8 for every new Neodymium compound that has been synthesized in the present study.

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**Conflict of Interest**

There is no conflict of interest associated with this work.

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