ELECTRONIC SPECTRAL CHARACTERIZATION AND ANTIBACTERIAL ACTIVITY OF LANTHANIDE (III) - CEFTAZIDIME COMPLEXES

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Abstract:
The present work describes the spectrochemical study of some Lanthanide (III) - ceftazidime (Ln(III)-CEFZ) complexes. The impact of the ligand environment on electronic spectrum of the lanthanide ion with special reference to complexation and nature of Ln-ligand bond has been examined. The energy and intensity parameters for the complexes have also been evaluated. The ligand and all prepared complexes were screened for their antibacterial activities against Escherichia coli, Pseudomonas aeruginosa and Staphylococcus aureus and the results showed that antibacterial activity of ligand on complexation have been enhanced.

Keywords: Lanthanide; Ceftazidime; Electronic Spectrum; Antibacterial Activity.

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1. Introduction

Cephalosporins antibiotics have been effectively used in various bacteria infected diseases because of the presence of β-lactam ring [1, 2]. The Cephalosporin class of antibiotics is classified into four generations, ceftazidime is a third-generation cephalosporin which demonstrates good activity against gram-negative organisms and some gram positive organism. It is also effective for biliary-tract infections, bone and joint infections, cystic fibrosis (respiratory-tract infections), endophthalmitis, infections in immune compromised patients (neutropenic patients), meningitis, peritonitis, pneumonia, sepsicemia, skin infections (including burns, ulceration) and urinary-tract infections [3]. Various strategies have been made to enhance the activity, or broaden the spectrum of these drugs, but the question about cost and adequacy of the supply, necessitate the use of novel agents [4]. For this purpose, thousands of compounds have been prepared based on well conceived ideas of improving their efficacy and have been subsequently screened but few of them were successfully passed the clinical tests [5, 6]. Some metal chelates of Ceftazidime and their antibacterial activity were evaluated Anacona [7] and Muslu [8]. The antibacterial activity of the metal complexes was found to be lower than that of free ceftazidime.
Lanthanide complexes is appealing in bioinorganic and coordination chemistry, because lanthanide ions have a tendency to form complex with coordination number greater than six, even up to twelve because of their size and charge [9]. The most important property of the chelating agents is its binding capacity which alters the behavior of resultant complex in lanthanide chelate complex and this bonding leads to improve the bio distribution and excretion profile of the lanthanide ions [10]. The spectroscopic properties of lanthanide(III) complexes with schiff base ligands has been studied by Koen Binnemans et al. [11] The chemical binding of Pr(III) complexes with L-alanyl-L-glutamine (Ala-Gln) and N-acetyl-L-glutamine (NAG) have been carried out in aqueous and different aquated organic solvents and their equimolar mixtures by using absorption difference and comparative absorption spectroscopy involving 4f-4f transitions spectra as probe [12]. Jorge Henrique Santos Klier Monteiro et al. [13] have determined the Judd-Ofelt intensity parameters of Sm(III) complexes. Judd–Ofelt Intensity Parameters and Spectral Properties of Gd₂O₃:Eu³⁺Nanocrystals have been studied by Chunxu Liu et al. [14]. A continued research work has been dedicated to lanthanide complexes, due to their successful application as diagnostic tools in biomedical analysis as MRI contrast agents [15]. Lanthanide complexes are effectively used as clinical diagnostic tool for cerebrospinal diseases and in assessment of central nervous system [16]. The lanthanide complexes have diverse biological activities, such as antioxidant activity, cytotoxic activity, DNA binding affinity [17, 18]. Lanthanide complexes with hesperetin hydrazones have been synthesized as a probe to antioxidants and therapeutic agents for some diseases [19]. Lanthanide complexes with Cloxacillin [20] and Morin [21] have also been synthesized as a probe to antimicrobial agents. Gadolinium (III) complexes are currently used for magnetic resonance imaging [22]. Lanthanide complexes especially samarium complexes have used as catalyst in selective hydroboration [23] as electroluminescent material [24], antimicrobial agents [25, 26] anticancer and anti proliferative agents [27, 28]. In this sequence, in the present work, we have studied the physiochemical, spectral and antimicrobial properties of Lanthanide (III)-Ceftazidime complexes.

2. Materials and Methods

All chemicals (reagents and solvents) were of reagent grade and used without any further purification. The ligand (Ceftazidime) was purchased from Scott-Edil Pharmacia Ltd. Double beam spectrophotometer (model 2203) was used to determine the UV-Vis spectra of complexes. All Ln(III)-CEFZ complexes were prepared using method adopted by earlier workers [29]. 0.01 M solutions of ligand and lanthanides were prepared in dichloro methane, and mixed in 1:1, 1:2, 1:3 and 1:4 Ln-ligand ratios. The pH of these systems was kept in between 6 and 7 by gradual addition of dil. NaOH or dil HCl immediately after mixing the Lantanide ion and ligand solution. Thus prepared Ln-ligand solutions were kept for one hour to attain equilibration. The spectra of all solutions were recorded in UV-Visible region.

For antibacterial study of ligand and complexes, the disc diffusion method was adopted [30]. For this, 200mL of Nutrient Agar Media was prepared and autoclaved for 30min at 120°C and 15 lbs pressure. 15mL of Nutrient Agar Media was poured in sterile Petri dishes and allowed to solidify. The test organisms were grown on agar medium in Petri dishes. The test compounds were added drop wise to a 5mm diameter filter paper paper disk, placed at the centre of the each Petri dish. The plates were incubated for 24 h at 36°C. The inhibition zone was evaluated after 24 h.
3. Result and Discussion

It has been found in observations that the complexes with 1:2 lanthanide-ligand ratio [31, 32], is the most favourable condition for the formation of lanthanide-Ceftazidime complexes. Therefore, all the spectral studies have been done on complexes with 1:2 M-L stoichiometry.

Electronic Spectra

Electronic spectra of prepared complexes were recorded to assess the covalency in M-L complexes. Typical spectral data obtained for solution of Ln (Ln= Pr, Nd, Sm and Ho) and CEFZ complexes is given in Table 3. The absorption bands appear in the spectra of Pr(III), Nd(III), Sm(III) and Ho(III) are due to transitions of e from the ground states $^3\text{H}_4$, $^4\text{I}_{9/2}$, $^6\text{H}_{5/2}$ and $^5\text{I}_8$ to the excited states i.e, J-levels of 4f$^n$-configuration [20, 33]. The treatment of results obtained in the spectral studies, was based on the calculation of various intensity and energy parameters as well as the application of various theories proposed for the spectral behaviour of lanthanide complexes.

Intensity parameters

These parameters incorporate oscillator strength and Judd-ofelt intensity parameters.

The observed oscillator strength is given by the expression:

$$P_{\text{obs}} = 4.6 \times 10^{-9} \times \varepsilon_{\text{max}} \times \Delta \nu_{1/2}$$

Where $\varepsilon_{\text{max}}$ is the molar extinction coefficient of the peak maximum and $\Delta \nu_{1/2}$ is half intensity band width.

The calculated oscillator strength is represented in terms of $T_2$, $T_4$ and $T_6$ parameters as given below:

$$P_{\text{cal}} = [T_2 [U^2]^2 + T_4 [U^4]^2 + T_6 [U^6]^2] \varpi$$

The values of $[U^2]^2$, $[U^4]^2$ and $[U^6]^2$ have been taken as reported by Carnall et. al. [34].

The Judd-Ofelt intensity parameters ( $T_\lambda$, $\lambda = 2, 4$ and $6$ ) are empirical, yet values are affected by minor changes in the coordination environment and symmetry of the molecule [35]. These parameters have been broadly utilized as a part of the structure elucidation of lanthanoid coordination compounds in solution [36]. The values of these three parameters ($T_2$, $T_4$ and $T_6$), evaluated by computational method, are given in Table 1.

| Complex | $T_2 \times 10^9$ (cm$^{-1}$) | $T_4 \times 10^9$ (cm$^{-1}$) | $T_6 \times 10^9$ (cm$^{-1}$) | $T_4/T_6$ | $\sigma_{\text{rms}} \times 10^6$ |
|---------|-----------------|-----------------|-----------------|----------|-----------------|
| Pr      | -27.502         | 0.67458         | 3.6064          | 0.1870   | 0.1842          |
| Nd      | 0.3438          | 0.6379          | 0.6845          | 0.9319   | 1.9264          |
| Sm      | 25.5230         | 1.5941          | 0.9001          | 1.7710   | 2.1982          |
| Ho      | 0.1460          | 0.2478          | 0.2720          | 0.9109   | 0.5321          |

For Pr (III) systems, $T_2$ value was found negative. This might be because of overlapping of f-d orbital in the region of f-f transition or due to using incorrect value of reduced matrix elements of
Pr (III) [37]. Table 1 is showing an appreciable variation in all the three $T_\lambda$ parameters ($T_2$, $T_4$ and $T_6$). The variation of $T_2$, $T_4$, $T_6$ parameters clearly indicate the high sensitivity towards coordination and symmetry changes. There are small changes observed in the value of $T_4/T_6$ ratio which suggest that symmetry changes are less prominent. The low value of $\sigma_{rms}$ deviation sows the applicability of Judd-Ofelt theory [38].

**Energy parameters**

These parameters give valuable data with respect to interelectronic repulsion, spin-orbit interaction, nephelauxetic effect and bonding in complexes. Energy parameters consist of following parameters:

**Slator-Condon $F_k$ ($k = 0$, 2, 4, 6):** The Slator integrals [39] are expressed in terms of Slator-Condon (Table-2) parameters ($F_k$) by following relations:

\[
\begin{align*}
F_2 &= F^2/225 \\
F_4 &= F^4/1089 \\
F_6 &= F^6/7361.64
\end{align*}
\]

**Lande parameters ($\zeta_{4f}$):** The correction factors $\Delta F_2$, $\Delta F_4$, $\Delta F_6$ and $\Delta \zeta_{4f}$ are obtained by employing the least squares fit method. The Slator-Condon ($F_2$, $F_4$, $F_6$) and the spin orbit $\zeta_{4f}$ parameters, obtained from following relation [29] (Table-2).

\[
\begin{align*}
\Delta F_2 &= F_2 - F_2^\circ \\
\Delta F_4 &= F_4 - F_4^\circ \\
\Delta F_6 &= F_6 - F_6^\circ \\
\Delta \zeta_{4f} &= \zeta_{4f} - \zeta_{4f}^\circ
\end{align*}
\]

where $F_2^\circ$, $F_4^\circ$, $F_6^\circ$ and $\zeta_{4f}^\circ$ are the zero order parameters.

**Racah parameters $E^k$ ($k = 1$, 2, 3):** The electrostatic interaction is computed in respect of Racah parameters (Table 2). These are the linear combination of $F_k$'s. They might be ascertained by following relations [40]:

\[
\begin{align*}
E^1 &= (70F_2 + 231F_4 + 2002F_6)/9 \\
E^2 &= (F_2 - 3F_4 - 7F_6)/9 \\
E^3 &= (5F_2 + 6F_4 - 91F_6)/3
\end{align*}
\]

| Complex | Racah Parameters ($E^k$) | Slater-Condon Parameters ($F_k$) | Lande Parameter $\zeta_{4f}$ |
|---------|---------------------------|---------------------------------|-----------------------------|
| Pr (III) | 4838.602 12.646 457.773 | 305.143 51.886 5.095 | 732.565 |
| Nd (III) | 4794.368 15.783 493.733 | 321.771 49.082 4.639 | 863.299 |

**Nephelauxetic ratio ($\beta$):** In the electronic spectra of the complexes, spectral bands show a shift towards lower energy, this may be due to the nephelauxetic effect [41]. The nephelauxetic ratio $\beta$ is calculated by using the following expression:
The value of $\beta$ is used to determine the covalency factor ($b^{1/2}$), Sinha parameter i.e. degree of metal–ligand covalency ($\delta\%$) and the covalency angular overlap parameter ($\eta$) by the following relations [42]:

$$b^{1/2} = [(1 - \beta)/2]^{1/2}$$

$$\delta\% = (1 - \beta) \ast 100/ \beta$$

$$\eta = 1 - \frac{b^{1/2}}{\beta^{1/2}}$$

The values of these parameters are present in table-3.

**Table 3: Electronic spectral data and related bonding parameters of Ln(III)-CEFZ complexes**

| Ln$^{3+}$ | Ln(NO$_3$)$_3$ Electronic spectral bands (cm$^{-1}$) | Electronic spectral bands of complexes (cm$^{-1}$) | J-levels | $\beta$ | $b^{1/2}$ | $\delta\%$ | $\eta$ |
|-----------|-----------------------------------------------|-----------------------------------------------|----------|----------|----------|----------|--------|
| Pr$^{3+}$ | 22545 22472 20833 16949 | 22396 21253 20682 16792 | $^3$H$_4 \rightarrow ^3$P$_2$ $^3$P$_1$ $^3$P$_0$ $^3$D$_2$ | 0.9934 0.9457 0.9927 0.9907 | 0.0574 0.1646 0.0600 0.0679 | 0.6634 5.7307 0.7275 0.9312 | 0.0033 0.0282 0.0036 0.0046 |
| Nd$^{3+}$ | 20188 19338 17510 14644 13812 12887 11921 | 19493 19047 17152 14577 13351 12468 11507 | $^4$I$_{9/2} \rightarrow ^4$G$_{9/2}$ $^4$G$_{7/2}$ $^4$G$_{5/2}$ $^4$F$_{9/2}$ $^4$F$_{7/2}$ $^4$F$_{5/2}$ $^4$F$_{3/2}$ | 0.9655 0.9849 0.9795 0.9954 0.96663 0.9675 0.9653 | 0.1311 0.0866 0.1010 0.0477 0.1291 0.1273 0.1316 | 3.5644 1.5245 2.0833 0.4578 3.4518 3.3537 3.5934 | 0.0176 0.0075 0.0103 0.0022 0.0171 0.0166 0.0178 |
| Sm$^{3+}$ | 29000 27933 26624 25825 24850 22870 21600 21450 | 28992 27624 26619 24987 24539 22857 21505 21110 | $^6$H$_{11/2} \rightarrow ^6$H$_{11/2}$ $^6$D$_{3/2}$ $^6$P$_{7/2}$ $^6$P$_{5/2}$ $^6$G$_{9/2}$ $^4$I$_{13/2}$ $^4$M$_{15/2}$ | 0.9997 0.9889 0.9998 0.9675 0.9875 0.9994 0.9956 0.9841 | 0.0116 0.0743 0.0095 0.1273 0.0789 0.0167 0.0468 0.0889 | 0.0268 1.1174 0.0183 3.3516 1.2637 0.0562 0.4400 1.6086 | 0.0001 0.0055 0.0001 0.0166 0.0062 0.0002 0.0021 0.0080 |
| Ho$^{3+}$ | 27594 24120 22240 21500 20800 18920 15860 | 27548 24038 22163 21133 20606 18660 15615 | $^5$I$_{8} \rightarrow ^5$G$_4$ $^5$G$_3$, $^3$G$_5$ $^5$G$_6$ $^3$F$_2$ $^5$F$_3$ $^5$F$_4$ $^3$F$_5$ | 0.9983 0.9966 0.9965 0.9829 0.9907 0.9863 0.9846 | 0.0288 0.0411 0.0416 0.0924 0.0683 0.0829 0.0878 | 0.1662 0.3392 0.3469 1.7380 0.9424 1.3923 1.5674 | 0.0008 0.0017 0.0017 0.0087 0.0047 0.0069 0.0078 |
For all complexes, $F_k$ parameters follow the order: $F_2 > F_4 > F_6$ and $E^k$ values follow the order $E^1 > E^3 > E^5$, which are in agreement with the finding of earlier workers [43]. There is a small decrease in value of $F_k$, $E^k$ and $\zeta_4$ parameters as compared to corresponding parameters of the free ion which suggesting the expansion of the central metal ion orbital on complexation [44]. The values of $\beta$ are found to be less than unity and the values of the covalency factor $b^{1/2}$ and Sinha’s parameter $\delta%$ are found to be positive in these complexes which is indicating covalent characters in the lanthanide–ligand bonding [45].

**Antibacterial Activity**

Antibacterial activity of the ceftazidime and their corresponding lanthanide complexes were carried out on selected three bacteria namely Escherichia coli, Pseudomonas aeruginosa and Staphylococcus aureus. The results of antibacterial activity are presented in Table 4, which shows that the ligand has strong activity against E. coli with inhibitory zones within 10–15 mm and moderate activity against P. aeruginosa with inhibitory zones within 5-10 mm, while the same ligand was found to be resistant against the gram positive bacteria, S. aureus.

The Pr(III) and Nd(III) complexes show equal or better activity compared to the ligand against the Gram-negative bacteria E. coli. The Sm(III) and Ho(III) complexes display moderate activity against the bacteria. All the complexes show moderate activity against Pseudomonas aeruginosa. All the complexes were found almost resistant against the gram positive bacterial species Staphylococcus aureus. From the data it is clear the metal complex shows enhanced antibacterial activities than that of the free ligand molecule. The activity of any compound is a complex combination of steric, electronic and pharmacokinetic factors [46]. The enhanced activity of the complexes is being elucidated by chelation behavior. This chelation increases the lipophilicity of the central atom through decreases polarity of metal ion in complexes. As a result the complexes obtained will be more soluble in the lipid Layer of cell membrane and show higher antibacterial activity [47].

| Compounds                  | E.c. | P.a. | S.a. |
|----------------------------|------|------|------|
| HL                         | +++  | ++   | -    |
| Pr(CEFZ)(H$_2$O)$_4$       | +++  | ++   | -    |
| Nd(CEFZ)(H$_2$O)$_4$       | +++  | ++   | -    |
| Sm(CEFZ)(H$_2$O)$_4$       | ++   | ++   | -    |
| Ho(CEFZ)(H$_2$O)$_4$       | ++   | ++   | -    |

Key to interpretation: E.c. – Escherichia Col; P.a. – Pseudomonas aeruginosa; S.a. – Staphylococcus aureus; (−) = no inhibition zone = inactive; 1–5 mm (+) = less active; 5–10 mm (++) = moderately active; 10–15 mm (++++) = highly active.

**4. Conclusion**

It can be concluded from these test results
- Interaction between Ln(III) ions and Ceftazidime is determined spectroscopically using 4f-4f transitions.
- The variation in the magnitude of energy interaction and intensity parameters shows that minor coordination changes in the Ln(III) complexes are caused by the different
coordinating sites of CEFZ, coordination number, denticity and nature of Ln(III)-CEFZ bond which do induce significant variation in the intensity of f-f transitions.

- The nephelauxetic ratio (β) and bonding parameters (b1/2) also describes the nature of the lanthanide-ligand bonding.
- The complexes are biologically active and show enhanced antibacterial activities compared to free ligand.

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