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 lantanide 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.*

1. Introduction

Cephalosphorins antibiotics have been effectively used in various bacteria infected diseasesbecause of the presence of β-lactam ring [1, 2]**.** The Cephalosphorin 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, septicaemia, 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 Gd_2O_3:Eu^{3+}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 prepeared 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 $120^{o}C$ and 15 Ibs 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 disk, placed at the centre of the each Petri dish. The plates were incubated for 24 h at 36ºC. 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 $^3H_4$, $^4I_{9/2}$, $^6H_{5/2}$ and $^5I_8$ 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:

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

Where ε_{max} is the molar extinction coefficient of the peak maximum and $\Delta\upsilon_{1/2}$ 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:

$$P_{cal} = [T_2 [U^2]^2 + T_4 [U^2]^2 + T_6 [U^6]^2] \bar{\upsilon}$$

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

The Judd-Ofelt intensity parameters (T_λ, $\lambda = 2, 4$ 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.

Table 1: Judd Oflet Parameters T_λ for Ln(III)-CEFZ Complexes

Complex	$T_2 \times 10^9$(cm)	$T_4 \times 10^9$(cm)	$T_6 \times 10^9$(cm)	T_4/T_6	$\sigma_{rms} \times 10^6$
Pr	-27.502	.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_λ 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 σ_{rms} deviation sows the applicability of Judd-Ofelt theory [38].

Energy parameters
These parameters give valuable data with respect to interelectronic repultion, 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:

$$F_2 = F^2/225$$
$$F_4 = F^4/1089$$
$$F_6 = F^6/7361.64$$

Lande parameters (ζ_{4f}): The correction factors ΔF_2, ΔF_4, ΔF_6 and $\Delta\zeta_{4f}$ are obtained by employing the least squares fit method. The Slater-Condon (F_2, F_4, F_6) and the spin orbit ζ_{4f} parameters, obtained from following relation [29] (Table-2).

$$\Delta F_2 = F_2 - F_2^\circ$$
$$\Delta F_4 = F_4 - F_4^\circ$$
$$\Delta F_6 = F_6 - F_6^\circ$$
$$\Delta\zeta_{4f} = \zeta_{4f} - \zeta^\circ_{4f}$$

where F_2°, F_4°, F_6° and ζ°_{4f} 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]:

$$E^1 = (70F_2 + 231F_4 + 2002F_6)/9$$
$$E^2 = (F_2 - 3F_4 - 7F_6)/9$$
$$E^3 = (5F_2 + 6F_4 - 91F_6)/3$$

Table 2: Energy parameters of Ln(III)-CEFZ complexes

Complex	Racah Parameters (E^k)			Slater-Condon Parameters (F_k)			Lande Parameter
	E^1	E^2	E^3	F_2	F_4	F_6	ζ_{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 (β): 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 β is calculated by using the following expression:

$$(1- \beta) = (\nu_{aquo} - \nu_{complex})/ \nu_{aquo}$$

The value of β 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 (η) by the following relations [42]:

$$b^{1/2} = [(1- \beta)/2]^{1/2}$$
$$\delta\% = (1- \beta)*100/ \beta$$
$$\eta = 1- \beta^{1/2}/\beta^{1/2}$$

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	β	$b^{1/2}$	$\delta\%$	η
Pr^{+3}	22545	22396	$^3H_4 \rightarrow ^3P_2$	0.9934	0.0574	0.6634	0.0033
	22472	21253	$\rightarrow ^3P_1$	0.9457	0.1646	5.7307	0.0282
	20833	20682	$\rightarrow ^3P_0$	0.9927	0.0600	0.7275	0.0036
	16949	16792	$\rightarrow ^1D_2$	0.9907	0.0679	0.9312	0.0046
Nd^{+3}	20188	19493	$^4I_{9/2} \rightarrow ^4G_{9/2}$	0.9655	0.1311	3.5644	0.0176
	19338	19047	$\rightarrow ^4G_{7/2}$	0.9849	0.0866	1.5245	0.0075
	17510	17152	$\rightarrow ^4G_{5/2}$	0.9795	0.1010	2.0833	0.0103
	14644	14577	$\rightarrow ^4F_{9/2}$	0.9954	0.0477	0.4578	0.0022
	13812	13351	$\rightarrow ^4F_{7/2}$	0.96663	0.1291	3.4518	0.0171
	12887	12468	$\rightarrow ^4F_{5/2}$	0.9675	0.1273	3.3537	0.0166
	11921	11507	$\rightarrow ^4F_{3/2}$	0.9653	0.1316	3.5934	0.0178
Sm^{+3}	29000	28992	$^6H_{5/2} \rightarrow ^4H_{7/2}$	0.9997	0.0116	0.0268	0.0001
	27933	27624	$\rightarrow ^4D_{3/2}$	0.9889	0.0743	1.1174	0.0055
	26624	26619	$\rightarrow ^6P_{7/2}$	0.9998	0.0095	0.0183	0.0001
	25825	24987	$\rightarrow ^6P_{3/2}$	0.9675	0.1273	3.3516	0.0166
	24850	24539	$\rightarrow ^6P_{5/2}$	0.9875	0.0789	1.2637	0.0062
	22870	22857	$\rightarrow ^4G_{9/2}$	0.9994	0.0167	0.0562	0.0002
	21600	21505	$\rightarrow ^4I_{13/2}$	0.9956	0.0468	0.4400	0.0021
	21450	21110	$\rightarrow ^4M_{15/2}$	0.9841	0.0889	1.6086	0.0080
Ho^{+3}	27594	27548	$^5I_8 \rightarrow ^5G_4$	0.9983	0.0288	0.1662	0.0008
	24120	24038	$\rightarrow ^5G_3, ^3G_5$	0.9966	0.0411	0.3392	0.0017
	22240	22163	$\rightarrow ^5G_6$	0.9965	0.0416	0.3469	0.0017
	21500	21133	$\rightarrow ^5F_2$	0.9829	0.0924	1.7380	0.0087
	20800	20606	$\rightarrow ^5F_3$	0.9907	0.0683	0.9424	0.0047
	18920	18660	$\rightarrow ^5F_4$	0.9863	0.0829	1.3923	0.0069
	15860	15615	$\rightarrow ^5F_5$	0.9846	0.0878	1.5674	0.0078

For all complexes, F_k parameters follow the order: $F_2 > F_4 > F_6$ and E^k values follow the order $E^1 > E^3 > E^2$, which are in agreement with the finding of earlier workers [43]. There is a small decrease in value of F_k, E^k and ζ_{4f} 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 β are found to be less than unity and the values of the covalency factor $b^{1/2}$ and Sinha's parameter δ% are found to be positive in these complexes which is indicating convalent 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 eluidated 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]**.**

Table 4: Antibacterial activity of Ceftazidime and its complexes against different bacteria

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 aeruguinosa; 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.

Acknowledgement

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