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STUDIES ON MIXED TERNARY YTTRIUM(III) COMPLEXES: PHYSICO-CHEMICAL INVESTIGATIONS, SPECTRAL CHARACTERIZATION, ANTIBACTERIAL, AND ANTIFUNGAL ACTIVITIES

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ABSTRACT

The mixed ligand Y(III) complexes were synthesized and characterized using advanced techniques. The primary ligand employed was 1-nitroso-2-naphthol, complemented by amino acids such as L-alanine, L-phenylalanine, and L-valine as secondary ligands. The complexes were examined using FTIR, UV-VIS spectroscopy, and thermal studies. Additional insights were obtained through elemental analysis, electrical conductivity, and magnetic susceptibility measurements. Antimicrobial evaluations against *Klebsiella pneumoniae* (ATCC 4352), *Bacillus cereus* (ATCC 6630), and *Candida albicans* (ATCC 10231) were performed using *in vitro* techniques with the agar well diffusion method. It was discovered that the complexes were non-electrolytic in dimethylformamide at a concentration of 10^{-3} M. The coordination of metal ions through nitrogen and oxygen donor atoms was validated by FTIR measurements. The diamagnetic behavior found in magnetic susceptibility investigations and UV-VIS spectra analysis showed transitions that were suggestive of electron transfer between the ligand and the metal. The coordinated water molecules were concluded through thermal analysis. When the antimicrobial activities of complexes were evaluated using the CLSI M07-A11 and agar gel protocols, they showed moderate to considerable activity levels in comparison to the standard drugs.

Keywords: Yttrium, Mixed-Ligand Coordination, 1-Nitroso-2-Naphthol, Amino Acids, Biological Activity, Physicochemical Studies

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INTRODUCTION

Metal-ligand complexes play a crucial role in advanced scientific research due to their superior biological functions compared to traditional complexes.[1] Their enhanced activity opens doors to innovative applications across biomedicine, catalysis, and material science.[2] Mixed-ligand systems, in particular, are gaining attention for their diverse bioactivities.[3] The incorporation of multiple ligands within a single framework enables interactions with various biological targets, significantly boosting their therapeutic potential and making them valuable tools for modern applications. Mixed-ligand complexes have shown remarkable potential in addressing antimicrobial resistance, inhibiting cancer cell growth through diverse mechanisms, and reducing oxidative stress by neutralizing free radicals.[4] Their ability to regulate inflammation further enhances their therapeutic value. This versatility positions them as promising candidates for developing next-generation drugs.[5] Additionally, ligand-based metal complexes are effective as catalysts in critical chemical reactions, including those targeting biological systems and synthetic pathways.[6,7] Ternary complexes exhibit advanced material applications in electronics, magnetism, and photonics, owing to their unique properties.[8,9] Mixed-ligand complexes incorporating 1-nitroso-2-naphthol and amino acids stand out due to their adaptable structures, offering applications in drug delivery and improved solubility.[10]

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Stability is significantly enhanced in ternary complexes containing nitrogen and oxygen donor ligands, as the combination strengthens coordination bonds. Chiral complexes of 1-nitroso-2-naphthol and amino acids also demonstrate specific binding properties, useful for detecting metal ions and environmental cleanup.[11] This blend of structural adaptability and bioactivity highlights the transformative potential of these complexes across multiple disciplines.[12] In the present work, we aim to synthesize mixed ligand ternary complexes of Y(III) with L-alanine, L-phenylalanine, and L-valine serving as secondary ligands and 1-nitroso-2-naphthol as the principal ligand for their biological assessment.

EXPERIMENTAL

Materials and Methods

All reagents and solvents were procured from industrial suppliers. Analytical reagent grade Yttrium(III) chloride hexahydrate was used from (Chemsworth), 1-nitroso-2-naphthol was purchased from (S. D. Fine Chemicals), and Amino acids L-alanine, L-phenylalanine and L-valine were procured from (Molychem, Mumbai). Solvents such as chloroform, methanol, dimethylsulfoxide (DMSO), ethanol, and dimethylformamide (DMF) were distilled and purified following established standard methods.[13]

Stepwise Approach to Yttrium Coordination Complex Formation

Chiral ternary complexes with mixed ligands were prepared through yttrium(III) chloride hexahydrate($YCl_3 \cdot 6H_2O$), 1-nitroso-2-naphthol (1N2N), and amino acids L-alanine, L-phenylalanine, and L-valine. A 10 mL aqueous solution of yttrium(III) chloride hexahydrate (0.372 g, 1 mmol) was combined with a 20 mL ethanol solution containing 1-nitroso-2-naphthol (0.346 g, 2 mmol) under continuous stirring. The preparation was stirred persistently for 10 minutes in a boiling water bath. Subsequently, a 1 mmol amino acid solution prepared in 10 mL of water was incorporated into the heated reaction mixture with constant stirring. The solution was maintained at 50°C in a water bath for heating. The ternary complexes were formed by adding a diluted form of ammonia solution to raise the pH level of the blend to 7.0. The consequent solution was set aside to cool to ambient temperature, and the final residue was washed off with water, and then ethanol. The complexes were separated by filtration and subsequently dried under vacuum.

Characterization Methods

The C, H, and N components were measured with a Vario Micro Cube Elemental Analyzer at the Chemistry Department, Delhi University. Gravimetric methods were applied to conclude the metal content.[14] Electronic spectra for the coordinated complexes were carried out in the 200 to 800 nm range using a Cary 5000 UV-Vis spectrophotometer (Agilent Technologies) and a quartz optical cell with a 10 mm path length. Spectra for FT-IR were carried out across the spectrum of 400 cm^{-1} to 4000 cm^{-1} using a Perkin-Elmer Spectrum I spectrometer, the samples were examined in the form of KBr pellets. The Shimadzu DTG-60H system was employed for thermogravimetric analysis and maintained a controlled heating rate of 10°C/min while raising the temperature from 25°C to 500°C under nitrogen flow. The electrical conductivity of the complexes was evaluated using 10^{-3} M solutions of the product in DMF at ambient conditions using a pw9527 Digital conductivity meter (Philips). The magnetic susceptibilities at room conditions were evaluated using the Guoy technique, with $Hg[Co(SCN)_4]$ serving as the calibration reference. The proposed structural design of the ternary complexes was elucidated using ChemDraw software.

Antimicrobial Screening

Agar Well Diffusion Method

The agar well diffusion method was utilized to assess the antimicrobial efficacy of the test compounds against selected microorganisms. This technique is particularly appropriate for evaluating semisolid and liquid samples and was systematically implemented in the study. Nutrient Agar plates inoculated with standardized cultures of *Klebsiella pneumoniae* and *Bacillus cereus* and Sabouraud Chloramphenicol Agar plates inoculated with *Candida albicans*, were prepared. Wells were developed in the agar using sterile tools, and 200 µl of a 5 mg sample dissolved in 500 µl DMSO was added to each well. The inoculated agar plates were kept at 37°C for bacterial cultures and 28°C for fungal cultures, with incubation periods of 24 hours and 48 hours, respectively. Zones of inhibition (ZOI) were observed to

assess antimicrobial activity. Miconazole and Ciprofloxacin were included as antifungal and antibacterial controls. The results provided insights into the bioactivity of the ternary metal complexes tested.

Minimum Inhibitory Concentration (MIC)

CLSI M07-A11 protocol was followed for (MIC). The selected test organisms included *Bacillus cereus* (ATCC 6630), *Klebsiella pneumoniae* (ATCC 4352), and *Candida albicans* (ATCC 10231) with initial inoculum levels of 2.60 × 10^6CFU/ml, 1.80 × 10^6CFU/ml, and 1.75 × 10^5CFU/ml, respectively. Agar media were tailored to the microbial types: Soybean Casein Digest Agar for bacterial strains and Sabouraud Chloramphenicol Agar for fungal strains. Sterile wells were made in the solidified agar using a cork borer, and test solutions were prepared in DMSO starting from a stock of 1 mg/ml. Serial dilutions of 50–1000 µg/ml were tested, with 200 µl of each concentration added to respective wells. The inoculated agar plates were kept at 37°C for bacterial cultures and 28°C for fungal cultures, with incubation periods of 24 hours and 48 hours, respectively. Post incubation, plates were assessed for inhibition zones to evaluate antimicrobial effectiveness.

RESULTS AND DISCUSSION

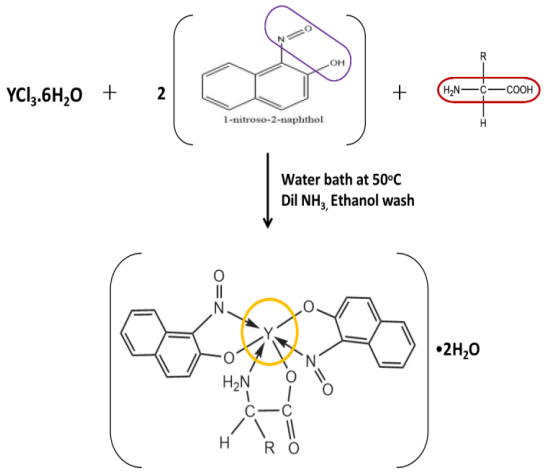
Reaction and Physical Properties of Metal Complexes

The synthesis of ternary mixed-ligand Y(III) coordinated complexes (Scheme-1) can be expressed as follows.

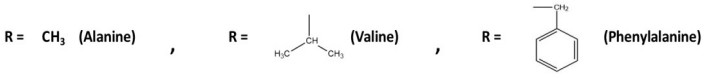
$$YCl_3.6H_2O + 2(1N2N) + HL \longrightarrow [\,Y\,(1N2N)_2\,(L)]\cdot 2H_2O + 3\,HCl + 4\,H_2O$$

Where, (1N2N) refers to 1-nitroso-2-naphthol while HL represents an amino acid.

Ternary mixed complexes are dark brown, non-hygroscopic, thermally stable solids, which suggests strong metal-ligand bonding (Table-1). The range of decomposition temperature of ternary yttrium(III) complexes was found in between 230-245°C (Table-2). Conventional organic solvents, namely ethanol, acetone, and others, do not effectively dissolve the complexes. However, they dissolve reasonably well in DMSO and DMF solvents.



Scheme-1



Physicochemical Characterization of Metal Complexes

The elemental analysis results, detailed in Table-1, confirm the formation of the expected 1:2:1 complex with the formula ([Y(1N2N)$_2$(L)]·2H$_2$O). Electrical conductivity measurements were performed in

dimethylformamide (10^{-3} M) concentration, revealing molar conductance values ranging from 0.000052 to 0.000056 Mhos cm^2 mol^{-1}. These values indicate a non-electrolytic nature for the complexes, as summarized in Table-2. Magnetic susceptibility measurements, corrected for diamagnetic contributions, further confirmed the diamagnetic properties of the ternary Y(III) complexes, as shown in Table-2. A 10^{-3} M concentration of dimethylformamide solution was used for the electronic absorption study in the UV-visible range in Table-3. The spectra show three transitions, namely π→π* at 282-292 nm, n→π* at 381-396 nm, and ligand to metal charge transfer transitions detected at 420-426 nm.[15]

Table-1: Physicochemical Profile of Yttrium(III) Mixed-Ligand Complexes

Complex	Empirical formula	Molecular weight	Elemental Profile %found(calculated)				Colour	pH
			%M	%C	%H	%N		
$[Y(1N2N)_2(Ala)]\cdot 2H_2O$	$C_{23}H_{22}N_3O_8Y$	557.35	15.89 (15.95)	49.50 (49.52)	3.91 (3.94)	7.50 (7.54)	Dark brown	7.01
$[Y(1N2N)_2(PhAl)]\cdot 2H_2O$	$C_{29}H_{26}N_3O_8Y$	633.45	14.01 (14.04)	54.90 (54.94)	4.08 (4.10)	6.61 (6.63)	Dark brown	7.00
$[Y(1N2N)_2(Val)]\cdot 2H_2O$	$C_{25}H_{26}N_3O_8Y$	585.40	15.10 (15.19)	51.22 (51.25)	4.41 (4.44)	7.12 (7.17)	Dark brown	6.95

Table-2: Physicochemical Profile of Yttrium(III) Mixed-Ligand Complexes

Complex	Decomposition temperature (°C)	Molar conductance (Mhos cm^2 mol^{-1})	μeff (B.M.)
$[Y(1N2N)_2(Ala)]\cdot 2H_2O$	230	0.00051	Diamagnetic
$[Y(1N2N)_2(PhAl)]\cdot 2H_2O$	245	0.00048	Diamagnetic
$[Y(1N2N)_2(Val)]\cdot 2H_2O$	235	0.00050	Diamagnetic

Table-3: Electronic Absorption and Transitions in Yttrium(III) Complexes

Complex	λ(nm)	ν(cm^{-1})
$[Y(1N2N)_2(Ala)]\cdot 2H_2O$	291	34364
	396	25253
	425	23529
$[Y(1N2N)_2(PhAl)]\cdot 2H_2O$	282	35461
	382	26178
	425	23529
$[Y(1N2N)_2(Val)]\cdot 2H_2O$	284	35211
	381	26247
	426	23474

Table-4: FTIR Spectroscopic Characterization of Yttrium(III) Complexes

S. No.	Complex	ν(O-H) H2O	ν(N-H) ASYM (AA)	ν(N-H) SYM (AA)	ν(C=O) (AA)	ν(N=O) (1N2N)	ν(C-O) (AA)	ν(C-O) (1N2N)	ν(C-N) (1N2N)	ν(C-N) (AA)	ν(Y-O)	ν(Y-N)
1	$[Y(1N2N)_2(Ala)]\cdot 2H_2O$	3386 (b)	3104(w)	3043 (w)	1609 (s)	1550 (s)	1396(w)	1152 (w)	1210 (m)	842(m)	635(w)	522(w)
2	$[Y(1N2N)_2(PhAl)]\cdot 2H_2O$	3415 (b)	3068(w)	3030 (w)	1611 (s)	1552 (s)	1396(w)	1149 (w)	1211 (m)	841(m)	633(w)	523(w)
3	$[Y(1N2N)_2(Val)]\cdot 2H_2O$	3409 (b)	3109(w)	3065 (w)	1610 (s)	1551 (s)	1403(w)	1148 (w)	1205 (m)	844(m)	636(w)	518(w)

AA = amino acids, 1N2N= 1-nitroso-2-naphthol, SYM = symmetric, ASYM = asymmetric, b = broad band, m = medium band, w = weak band, s = strong band

Fourier Transform Infrared Spectra

The FTIR analysis provides crucial insights into the structural characteristics and ligand coordination in Yttrium(III) complexes. Spectra recorded (Table-4) within 4000–400 cm^{-1} revealed significant shifts indicating ligand attachment. The absence of the O-H stretch around 3448 cm^{-1}, characteristic of free 1-nitroso-2-naphthol, confirms deprotonation and its role in complexation. The υ(CO) band shift to 1152–1148 cm^{-1} indicates bidentate coordination via nitrogen and oxygen. Peaks at 1557–1554 cm^{-1} and 1212–1211 cm^{-1} verify the presence of nitroso and nitrile groups, respectively. In amino acids, the N-H bands at 3040 cm^{-1} and 2960 cm^{-1} shift to 3117–3109 cm^{-1} and 3062–3048 cm^{-1}, evidencing amino group coordination. Chelated nitrile groups appeared at 840 cm^{-1}, while asymmetric and symmetric COO^- stretches at 1610–1606 cm^{-1} and 1401–1400 cm^{-1} confirmed carboxylic group binding. Broad bands at 3407–3387 cm^{-1} suggest coordinated water molecules. Weak peaks at 642–640 cm^{-1} and 522–520 cm^{-1} were attributed to Y-O and Y-N bonds, respectively, absent in free ligands, further affirming complex formation. Thermal studies supported these findings.[16]

Thermal Studies

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were employed at a steady rate of temperature increase of 10°C/min under a nitrogen-controlled environment to assess the decomposition behavior and stages of the synthesized Y(III) ternary complexes. The information gathered from this thermal analysis is summarized in Table-5. The thermogravimetric assessment of the Y(III) mixed-ligand complexes reveals varying degrees of thermal stability. As the temperature increases, the complexes undergo gradual weight loss due to decomposition triggered by fragmentation. The ternary complexes with L-alanine, L-phenylalanine, and L-valine exhibit similar behavior in both TG and DTA analyses. In the TG study, the initial weight loss noted across the temperature span of 70-95°C is associated with the removal of two ligand-bound water molecules. A subsequent diminution of weight in the 230-360°C range was because of the decomposition of the amino acid ligands, followed by another significant weight loss between 420-560°C, which reflects the decomposition of the two 1-nitroso-2-naphthol moieties. The DTA analysis exhibits an endothermic peak occurring between 75°C and 100°C, indicating the presence of crystallized water molecules. With additional temperature elevation, two defined exothermic regions are observed: a minor peak between 250°C and 400°C and a broader one from 430°C to 580°C. These exothermic peaks correspond to the decomposition of the amino acid ligands and the breakdown of the 1-nitroso-2-naphthol groups, respectively. After 500°C, the weight stabilizes, indicating the complete decomposition of the complexes into a fine metal oxide residue. The conversion was conclusively supported by XRD analysis of the decomposed material.

Table-5: Thermal Behavior of Yttrium(III) Complexes

S. No.	Complex	Temperature Range (°C)	Loss of mass due to	Mass Loss %	
				Found	Calculated
1.	$[Y(1N2N)_2(Ala)]\cdot 2H_2O$	70-90	Two water molecules	6.21	6.29
		230-340	Amino acid	16.35	16.61
		425-550	Two 1N2N molecules	77.38	77.43
2.	$[Y(1N2N)_2(PhAl)]\cdot 2H_2O$	75-91	Two water molecules	5.55	5.68
		245-355	Amino acid	27.45	27.65
		430-555	Two 1N2N molecules	80.05	80.12
3.	$[Y(1N2N)_2(Val)]\cdot 2H_2O$	72-95	Two water molecules	6.03	6.15
		235-350	Amino acid	21.20	21.32
		427-565	Two 1N2N molecules	80.02	80.12

Biological Studies

The antibacterial and antifungal activity of synthesized yttrium(III) complexes was observed by using the Agar Well Diffusion technique as per CSLI M07-A11 standards. The complexes were tested against *Bacillus cereus*, *Klebsiella pneumoniae*, and *Candida albicans*. The findings from the agar well diffusion assay indicate that the complexes demonstrate moderate sensitivity to *B. cereus* and *K. pneumoniae*, with better inhibition observed for *B.cereus* compared to *K.pneumoniae* (Table-6). The complex $[Y(1N2N)_2(PhAl)\cdot 2H_2O]$ demonstrated greater potency against *K.pneumoniae* than

$[Y(1N2N)_2(Ala)\cdot 2H_2O]$ and $[Y(1N2N)_2(Val)\cdot 2H_2O]$. Within the scope of this work, the minimum inhibitory concentration (MIC) for the various complexes was observed to be 0.2 mg/ml (Table-7), which is lower than that reported in previous research. For antifungal activity, all complexes demonstrated significant inhibition of *C. albicans*, with minimum inhibitory concentrations (MIC) observed at 0.2 mg/ml, aligning with previous studies (Table-8). The enhanced bioactivity is attributed to chelation, which increases the complex's hydrophobicity and facilitates penetration through microbial lipid layers.[17] These findings underscore the potential of yttrium complexes in combating bacterial and fungal infections, as shown in Fig.-4. From the perspective of the physicochemical investigations, the binding interactions and framework of the yttrium(III) chiral complexes are illustrated in Fig.-1 to 3.

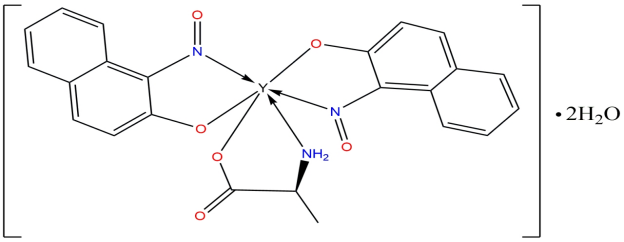


Fig.-1: Proposed Structure of $[Y(1N2N)_2(Ala)]\cdot 2H_2O$ Complex

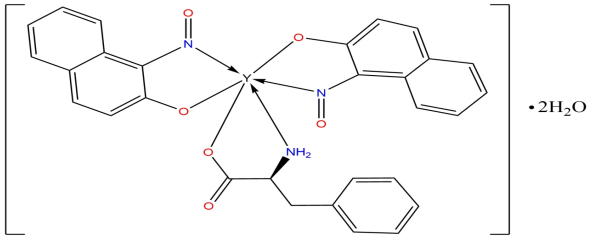


Fig.-2: Proposed Structure of $[Y(1N2N)_2(PhAl)]\cdot 2H_2O$ Complex

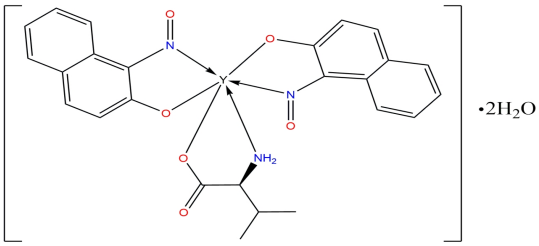


Fig.-3: Proposed Structure of $[Y(1N2N)_2(Val)]\cdot 2H_2O$ Complex

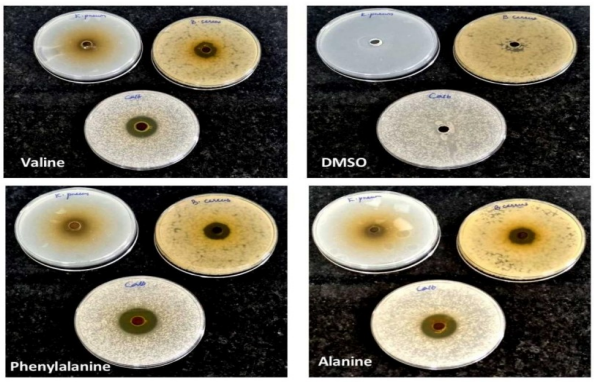


Fig.-4: Zone of Inhibition of *Klebsiella pneumoniae, Bacillus cereus,* and *Candida albicans* for the Synthesized Yttrium(III) Complexes

Table-6: Antibacterial Activity (Mm) of Yttrium Complexes Via Agar Well Diffusion Method

S. No.	Complex	Antibacterial Activity (mm) with	
		B.cereus	*K.pneumoniae*
1.	$[Y(1N2N)_2(Ala)]\cdot 2H_2O$	06	04
2.	$[Y(1N2N)_2(PhAl)]\cdot 2H_2O$	05	3.5
3.	$[Y(1N2N)_2(Val)]\cdot 2H_2O$	06	04
4.	Ciprofloxacin	25	14

Table-7: MIC Data of the Yttrium Complexes by Agar Well Diffusion Method for Antibacterial Activity

S. No.	Complex	MIC (mg/ml)	
		B.cereus	*K .pneumoniae*
1.	$[Y(1N2N)_2(Ala)]\cdot 2H_2O$	0.2	0.2
2.	$[Y(1N2N)_2(PhAl)]\cdot 2H_2O$	0.2	0.2
3.	$[Y(1N2N)_2(Val)]\cdot 2H_2O$	0.2	0.2

Table-8: Antifungal Activity (Mm) of the Yttrium Complexes by Agar Well Diffusion Method

S.No.	Complex	Antifungal Activity (mm)	MIC (mg/ml)
		C.albicans	*C.albicans*
1.	$[Y(1N2N)_2(Ala)]\cdot 2H_2O$	08	0.2
2.	$[Y(1N2N)_2(PhAl)]\cdot 2H_2O$	8.5	0.2
3.	$[Y(1N2N)_2(Val)]\cdot 2H_2O$	7.5	0.2
4.	Micanazole	20	-

CONCLUSION

The study provides several significant conclusions about the synthesized complexes. Thermal and XRD analysis confirmed the molecules of water in the chiral complexes. The spectral data indicate metal coordination through nitrogen and oxygen donor atoms, with FTIR confirming metal-ligand bonding. Elevated decomposition temperatures suggest strong metal-ligand interactions, supported by conductance measurements, which show non-electrolytic behavior with values below 1 mho cm^2 mol^{-1}. Magnetic studies, including the Gouy method, confirm the diamagnetic nature of the complexes. Additionally, in the recorded electronic absorption spectra, intraligand transitions, and charge transfer mechanisms are evident, and elemental analysis suggests a composition ratio of 1:2:1, with a coordination number of 6 proposed for these yttrium-mixed ligand complexes. Antibacterial assessments demonstrate that the complexes are moderately resistant to *Klebsiella pneumoniae, Bacillus cereus,* and *Candida albicans* showing average potency for antifungal activity when compared to the standard antibacterial agent ciprofloxacin and the standard antifungal agent miconazole.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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