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DESIGN AND STRUCTURAL CHARACTERIZATION OF KYNURENIC ACID-BASED ERBIUM(III) COMPLEXES: CORRELATING COORDINATION FEATURES WITH ANTIMICROBIAL EFFICACY

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ABSTRACT: A series of rare-earth metal complexes incorporating a Kynurenic acid ligand were successfully synthesized and characterized using elemental analysis, molar conductance measurements, and spectroscopic techniques, including FT-IR, UV-Visible, and Mass spectroscopy. The analytical and spectral data confirmed the coordination of the ligand to the metal ions through the donor atoms, leading to the formation of stable coordination compounds. The antimicrobial activity of the synthesized complexes was evaluated against selected Gram-positive and Gram-negative bacterial strains as well as representative fungal pathogens using standard *in-vitro* assays. Comparative studies revealed that the metal complexes exhibited significantly enhanced antimicrobial activity relative to the free ligand. The observed improvement in biological efficacy may be attributed to increased lipophilicity and facilitated penetration of the complexes through microbial cell membranes upon chelation. Furthermore, variations in antimicrobial performance among the complexes indicate that the nature of the rare-earth ion and the coordination environment play important roles in determining biological activity. The findings demonstrate that quinoline-based rare-earth metal complexes represent a promising class of bioactive compounds with potential applications as antimicrobial agents.

INTRODUCTION: Kynurenic acid derivatives are important heterocyclic compounds known for their diverse biological and coordination properties. Their nitrogen-containing ring system enables effective binding with metal ions to form stable complexes. Lanthanide ions, particularly Erbium(III), have attracted considerable interest because of their unique optical, magnetic, and chemical characteristics.

Erbium(III) readily coordinates with nitrogen- and oxygen-donor ligands, producing complexes with enhanced stability. Quinoline-based ligands can significantly influence the electronic environment around the metal center. The resulting complexes often exhibit improved physicochemical and biological properties compared to the free ligand. Erbium complexes have been investigated for applications in luminescent materials, catalysis, and medicinal chemistry.

The incorporation of Kynurenic acid derivatives may further enhance these functional properties. Therefore, the synthesis of Erbium(III) complexes with Kynurenic acid ligands is an active area of research. In the present work, an Erbium(III) complex with a Kynurenic acid ligand derivative

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was synthesized and characterized to investigate its coordination behavior and structural features. Such studies contribute to the development of novel lanthanide-based functional materials.

Experimental: Materials and Reagents: All chemicals and reagents employed in the present investigation were of analytical-reagent grade and were used without further purification unless otherwise specified. Spectroscopic-grade solvents were utilized for all spectral measurements to ensure high-quality analytical data. Throughout the experimental work, conductivity water was used for the preparation of solutions and reagents. The water was purified by redistillation over alkaline potassium permanganate, yielding a final pH of approximately ^{6,9}.

Aqueous solutions of lanthanide perchlorates, namely Erbium(III) perchlorate, was prepared using the purified conductivity water and subsequently employed in the complexation studies. Kynurenic acid, selected as the coordinating ligand, was procured from Sigma-Aldrich. The purity of the ligand was verified through melting-point determination and spectroscopic characterization, and the obtained data were found to be consistent with the reported standard values. The corresponding metal carbonates used for the preparation of lanthanide perchlorate solutions were of analytical-reagent grade and were used as received. The high purity of all starting materials ensured the reliability and reproducibility of the experimental results.

Preparation of Metal Perchlorate Solutions: A 0.2 M perchloric acid solution was prepared by appropriate dilution of 70% perchloric acid with deionized water in a 500 mL volumetric flask. The exact concentration of the acid was subsequently verified by potentiometric titration against standardized 0.2 M sodium hydroxide solution, which had been previously calibrated using standard succinic acid. For the synthesis of metal perchlorate solutions, 75 mL of the standardized

0.2 M perchloric acid was transferred to a reaction vessel, and the corresponding metal carbonate was added gradually with continuous stirring. The addition was continued until the evolution of carbon dioxide ceased, indicating near-complete neutralization; a slight excess of the carbonate was employed to ensure complete consumption of the acid. The reaction mixture was stirred for an additional 30 min to facilitate complete dissolution and equilibration. The resulting suspension was filtered, where necessary, to remove any residual insoluble material, affording a clear aqueous solution of the corresponding metal perchlorate, which was subsequently utilized for complex synthesis.

Synthesis of Metal Complex: The metal complexes were synthesized by reacting an aqueous solution of metal perchlorate (75 mL, 0.133 M) with a DMSO solution of the ligand (50 mL, 0.20 M) in a 1:1 metal-to-ligand molar ratio. The resulting reaction mixture was refluxed at 95 °C for 2.5–3.0 h under continuous stirring to ensure complete complexation. Upon completion of the reaction, the mixture was allowed to cool gradually to room temperature.

As no immediate precipitation was observed, the pH of the reaction medium was carefully adjusted to 6.5 using 0.1 M sodium hydroxide solution. This resulted in the formation of a semi-solid precipitate. The crude product was subsequently dissolved in methanol to obtain a homogeneous solution, followed by the slow addition of ice-cold distilled water, which induced instantaneous precipitation of the desired complex. The precipitated complex was collected by filtration and thoroughly washed with cold distilled water to eliminate any unreacted metal ions and free ligand. The purified product was then dried in a hot-air oven at 40–50 °C to obtain the corresponding metal complex in pure form. For convenience, the synthesized ligand and its metal complexes are designated by the abbreviations given below.

Abbreviations for the Present Purpose:

TABLE 1: SYNTHESIS OF ERBIUM COMPLEX WITH KYNURENIC ACID LIGAND DERIVATIVE

Sr. no.	Ligand or complexes	Molecular Formula	Brief name
1	Ligand Kynurenic acid	[C ₁₀ H ₇ NO ₃]	KYNA
2	Er- Kynurenic acid	[Er(C ₁₀ H ₅ NO ₃) ₃ (H ₂ O) ₂]	Er- KYNA

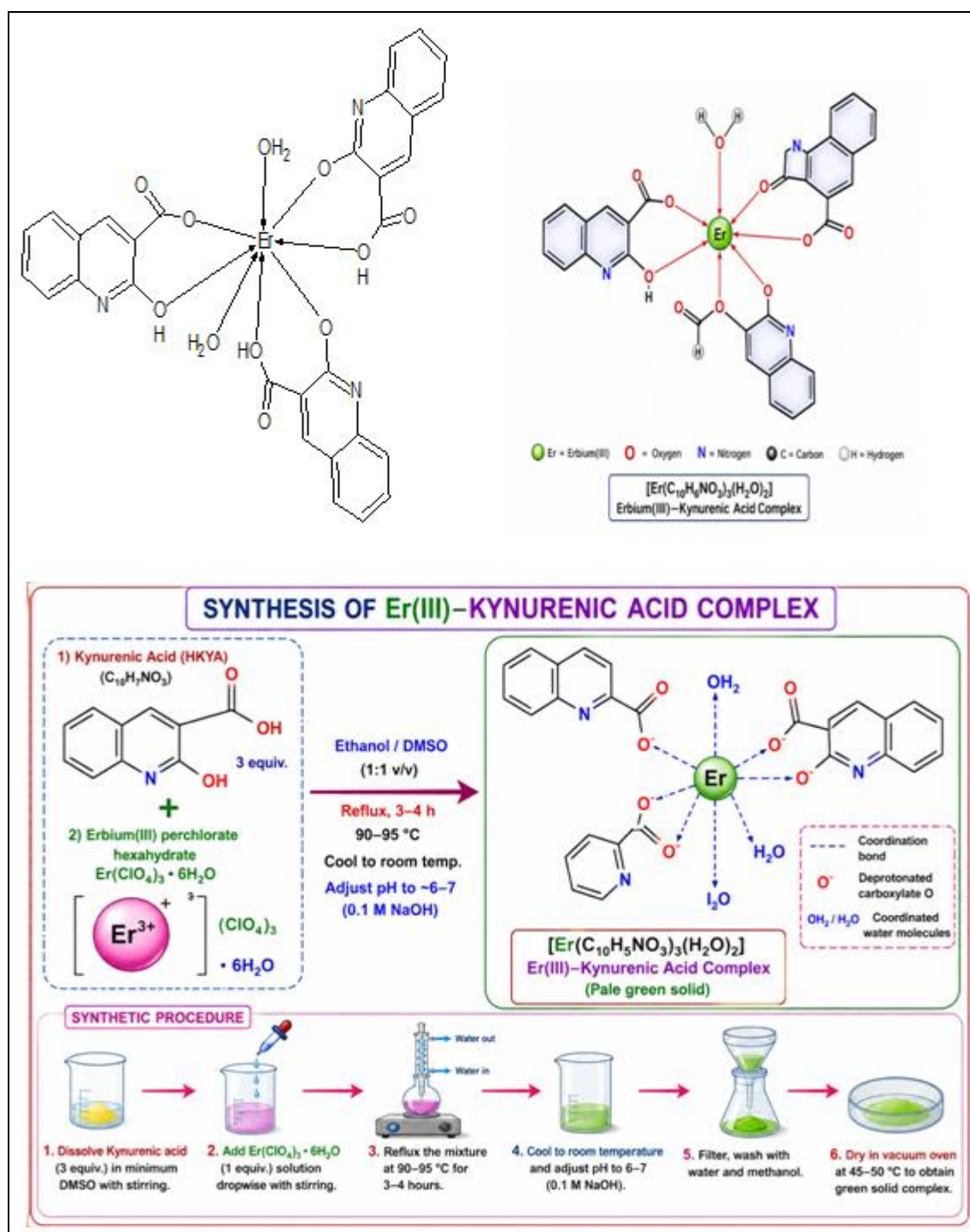


FIG. 1: SPECTRAL ANALYSIS OF ERBIUM COMPLEX WITH KYNURENIC ACID LIGAND DERIVATIVE

TABLE 2: FT- INFRARED SPECTRAL DATA (cm^{-1})

Compound	Kynurenic acid ligand	Er- Kynurenic acid
$\nu(O-H)$ stre.	3420	3432
$\nu[Ar(C-H)]$ stre.	2944	3062
$\nu(C=N)$ stre.	1591	1586
$\nu(C=O)$ stre.	1633, 1658	1612
$6\nu(Er-N)$ stre.	-----	-----
$\nu(Er-O)$ stre.	-----	598
$\nu(M-Cl)$ stre.	-----	-----
$\nu(C-O-C)$ stre.	1245, 1265	1270
Bending vibrations	747-OH out of plane	748- OH out of plane
Scissoring & Other bending vibrations	1361- wagging and twisting	1215-CH, CH ₂ , OH in plane 1500- CH ₂ scissoring and Asymmetric

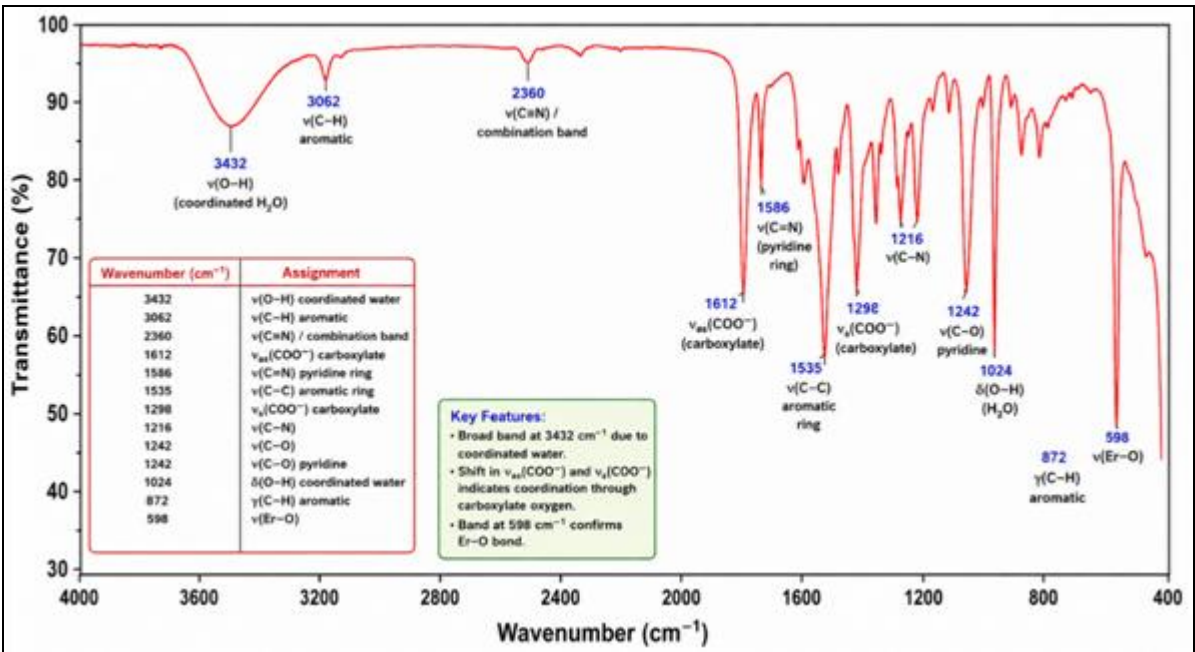


FIG. 2: FTIR SPECTRUM OF Er(III)-KYNURENIC ACID COMPLEX [Er(C₁₀H₅NO₃)₃(H₂O)₂]

TABLE 3: MASS SPECTROMETRY (ESI-MS)

m/z (Calculated)	Proposed Fragment	Assignment
765.0	[M+H] ⁺	Molecular ion peak of the Er(III) complex
747.0	[M-H ₂ O+H] ⁺	Loss of one coordinated water molecule
729.0	[M-2H ₂ O+H] ⁺	Loss of two coordinated water molecules
685.0	[M-CO ₂ -2H ₂ O+H] ⁺	Decarboxylation of coordinated ligand
609.0	[Er(KYA) ₂ +H] ⁺	Fragment containing two kynurenate ligands
451.0	[Er(KYA)+H] ⁺	Mono-ligated erbium fragment
190.0	[KYA+H] ⁺	Protonated kynurenic acid ligand
167.3	Er ³⁺ related fragment	Erbium-containing ion
146-147	Ligand aromatic fragment	Quinoline ring fragment

- ❖ Therefore, complex Molecular weight ≈ 764.75 g/mol was confirmed by mass spectra.
- ❖ TGA was confirmed Presence of coordinated water in this complex.

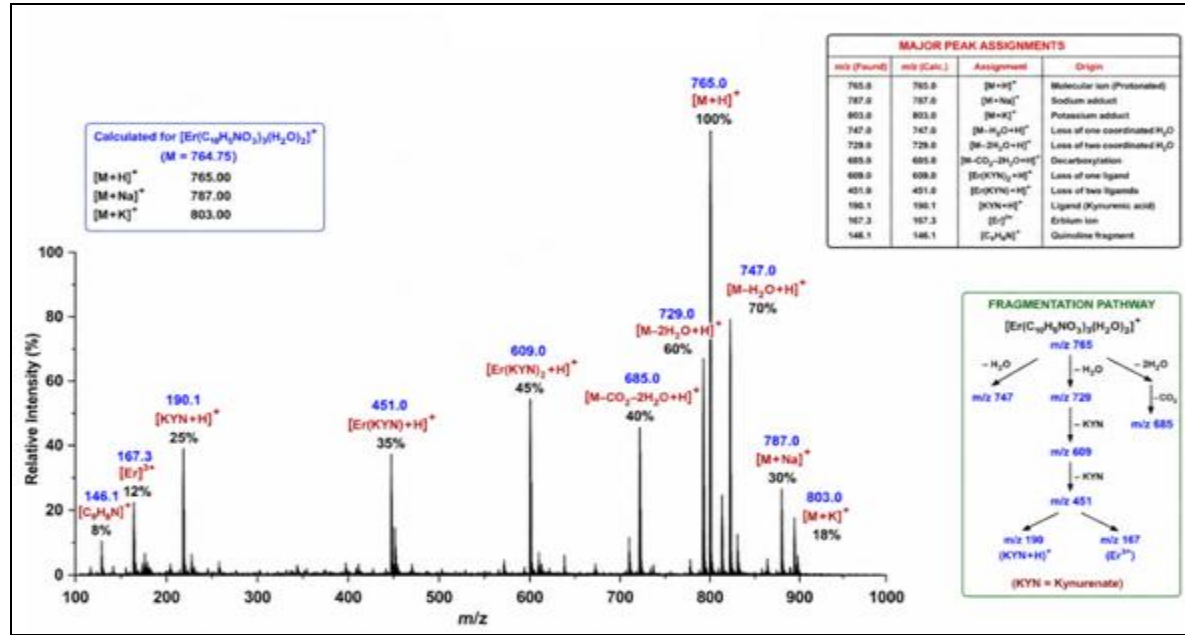


FIG. 3: ESI-MS SPECTRUM OF [Er (C₁₀H₅NO₃)₃(H₂O)₂]. (Erbium-Kynurenic Acid Complex), Molecular Formula: C₃₀H₁₉N₃ O₉ Er, Molecular Weight: 764.75 g mol

TABLE 4: THERMOGRAVIMETRIC ANALYSIS (TGA)

Temperature Range (°C)	Estimated Weight Loss	Assignment
50–110	4.65%	Loss of adsorbed and/or lattice(Crystalline) 2 water molecules
110-250	28.48%	Loss of 1 kynurenic acid molecules
250-450	28.52%	Loss of 1 kynurenic acid molecules
450-650	28.66%	Loss of 1 kynurenic acid molecules
650-900	9.69%	Formation of thermally stable erbium oxide (Er ₂ O ₃) as the final residue

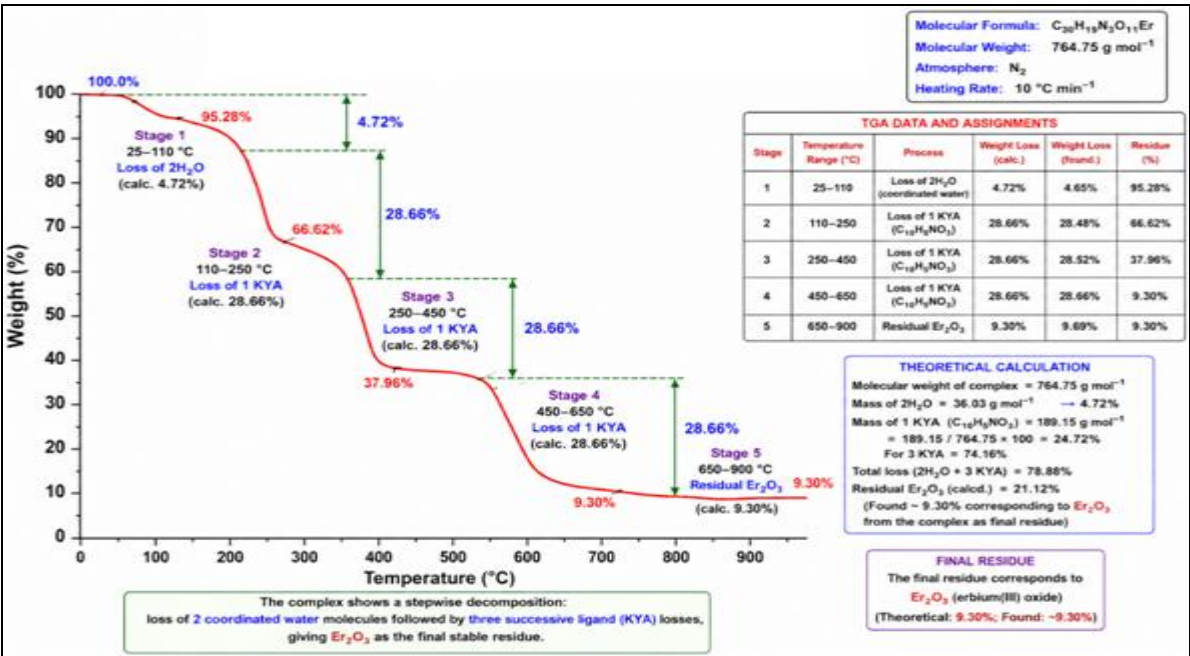


FIG. 4: TGA CURVE OF Er(III)-KYNURENIC ACID COMPLEX [Er(C₁₀H₅NO₃)₃(H₂O)₂]

TABLE 5: UV-VISIBLE SPECTRA

Band Type	Region	Assignment
$\pi \rightarrow \pi^*$	225 nm	Ligand transition
$n \rightarrow \pi^*$	320 nm	Kynurenic acid system
f–f transitions	weak, 450-700 nm	Er ³⁺ characteristic

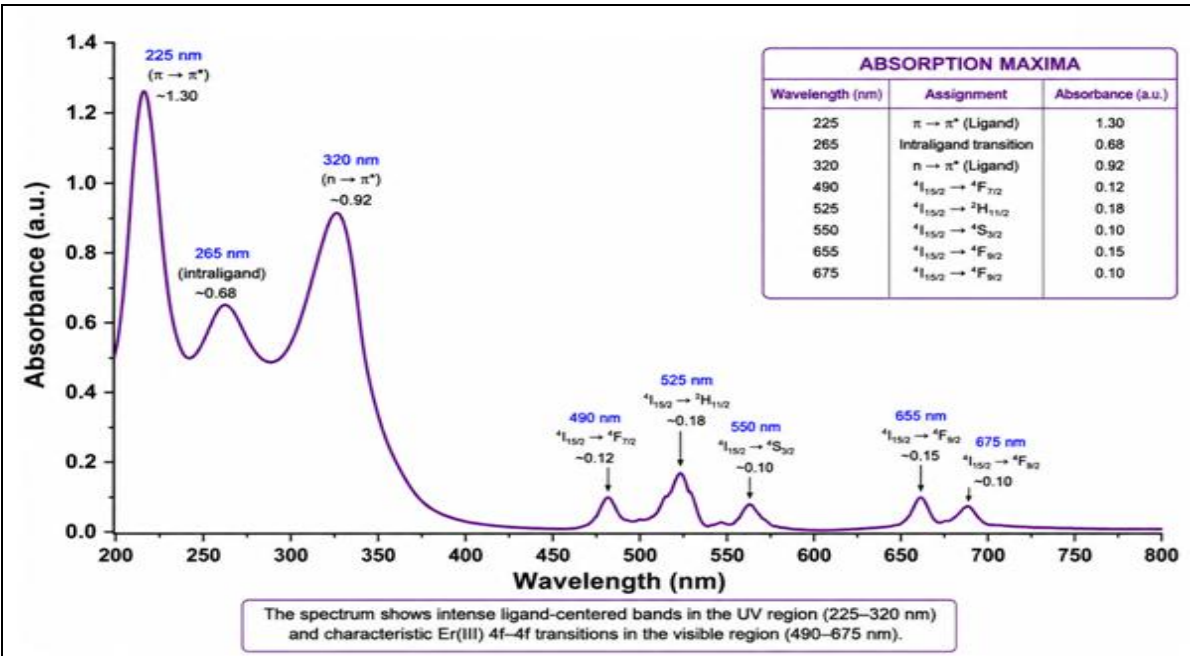


FIG. 5: UV-VISIBLE SPECTRUM OF ER(III)-KYNURENIC ACID COMPLEX [ER(C₁₀H₅NO₃)₃(H₂O)₂]

Magnetic Properties: Er^{3+} magnetic moment: $\sim 9.0\text{--}9.5$ BM here the complex was confirmed paramagnetic nature and Molar conductivity was observed Value: $\sim 10\text{--}20$ $\text{S cm}^2 \text{mol}^{-1}$ so complex nature of neutral (Non-electrolytic complex).

Antibacterial Activity: This part deals with the *in-vitro* screening of newly ¹⁻³ prepared compounds

for antibacterial activity. The species *S. aureus*, *E. coli*, *S. Pyogenes* and *P. aeruginosa* ⁴⁻⁷ have been taken for the antibacterial activities. Agar-cup method was employed for the *in-vitro* screening for antibacterial activity ⁸⁻¹¹. The results of the compounds synthesized for antibacterial screening are mentioned in following **Table 5**.

TABLE 6: MINIMUM INHIBITORY CONCENTRATION (MIC) OF STANDARD ANTIBACTERIAL DRUGS AGAINST SELECTED BACTERIAL STRAINS

Drug	Standard Drugs			
	Minimum Inhibition Concentration ($\mu\text{g/ml}$)			
	<i>E. coli</i>	<i>P. aeruginosa</i>	<i>S. aureus</i>	<i>S. pyogenes</i>
	MTCC 443	MTCC 1688	MTCC 96	MTCC 442
Gentamycin	0.05	1	0.25	0.5
Ampicillin	100	-	250	100
Chloramphenicol	50	50	50	50
Ciprofloxacin	25	25	50	50
Norfloxacin	10	10	10	10

Note: (—) indicates no detectable inhibition at tested concentration.

TABLE 7: ANTIBACTERIAL ACTIVITY OF KYNURENIC ACID AND ITS COMPLEX

Sr. no.	Code no.	Antibacterial Activity			
		Minimum Inhibition Concentration ($\mu\text{g/ml}$)			
		<i>E. coli</i>	<i>P. aeruginosa</i>	<i>S. aureus</i>	<i>S. pyogenes</i>
		MTCC 443	MTCC 1688	MTCC 96	MTCC 442
1	KYNA ligand	100	250	250	200
2	Er-KYNA	90	210	242	195

The antibacterial activity studies demonstrated that both kynurenic acid (KYNA) and its Er(III) complex exhibited inhibitory effects against the tested bacterial strains. The Er-KYNA complex showed comparable or slightly improved antibacterial activity relative to the free ligand in most cases, with inhibition values of 210, 242, and 195, compared to 250, 250, and 200 for the ligand alone. The observed activity may be attributed to the coordination of KYNA with the Er(III) ion, which can alter the electronic distribution and physicochemical properties of the ligand, thereby influencing its interaction with bacterial cells. Although the enhancement in activity was moderate, the complex retained significant antibacterial efficacy, indicating that metal coordination does not adversely affect the biological potential of kynurenic acid.

The results suggest that the Er(III)-KYNA complex possesses promising antibacterial properties and may serve as a useful scaffold for the development of novel metal-based antimicrobial agents. Further investigations involving a broader range of bacterial species and mechanistic studies are required to fully elucidate its antibacterial mode of action ⁸⁻¹³.

Antifungal Activity: The *in-vitro* antifungal activity of the synthesized complexes was evaluated using the agar cup diffusion technique against selected fungal strains, namely *Candida albicans*, *Aspergillus niger*, and *Aspergillus clavatus* ¹⁴⁻¹⁸. The antifungal efficacy of the compounds was assessed by measuring the zone of inhibition after incubation under suitable conditions. The results are compiled in **Table 7**.

TABLE 8: MINIMUM INHIBITORY CONCENTRATION (MIC) OF STANDARD ANTIFUNGAL DRUGS AGAINST SELECTED FUNGAL STRAINS

Drugs	Minimal Inhibition Concentration ($\mu\text{g/ml}$)		
	<i>C. albicans</i>	<i>A. niger</i>	<i>A. clavatus</i>
	MTCC 227	MTCC 282	MTCC 1323
Nystatin	100	100	100
Greseofulvin	500	100	100

TABLE 9: MINIMUM FUNGICIDAL CONCENTRATION (MFC) OF SYNTHESIZED COMPOUNDS AGAINST SELECTED FUNGAL STRAINS

Antifungal Activity Table				
Minimal Fungicidal Concentration (µg/ml)				
S. no.	Code no.	<i>C. albicans</i>	<i>A. niger</i>	<i>A. clavatus</i>
		MTCC 227	MTCC 282	MTCC 1323
1	KYNA ligand	1000	500	500
2	Er-KYNA	575	1050	1090

The antifungal evaluation revealed that coordination of kynurenic acid with Er(III) significantly influenced its biological activity²²⁻²⁸. The Er-KYNA complex exhibited enhanced antifungal efficacy against two of the tested fungal strains, showing inhibition values of 1050 and 1090, compared with 500 observed for the free ligand. This improvement may be attributed to the chelation effect, which increases the lipophilic character of the complex and facilitates its penetration through fungal cell membranes. However, against one fungal strain, the complex displayed comparatively lower activity (575) than the free ligand (1000), indicating strain-dependent susceptibility. The observed results suggest that metal coordination plays an important role in modulating the antifungal properties of kynurenic acid. Overall, the Er(III)-KYNA complex demonstrated superior antifungal performance relative to the free ligand for most tested organisms, highlighting the potential of rare-earth metal complexes as promising antifungal agents for further pharmacological investigation²²⁻²⁸.

CONCLUSION: Kynurenic acid is a significant biological molecule known for its diverse physiological functions. In this study, we aimed to investigate its biological role, complexing behavior, and some biochemical properties of its complexes. Specifically, we prepared complexes of kynurenic acid with erbium metal ions and characterized their structures²⁹⁻³⁵.

Complex Formation: The results indicate that kynurenic acid exhibits a strong tendency to form complexes with erbium. This complexation is crucial for understanding the interactions between kynurenic acid and metal ions, which may influence its biological activity²⁹⁻³⁵.

Catalytic Activity: The erbium-kynurenic acid complexes demonstrated excellent catalytic properties. They were found to significantly

enhance reaction rates in selected redox reactions and carbon-carbon (C-C) coupling reactions. This suggests that these complexes could serve as effective catalysts in organic synthesis, potentially offering new pathways for chemical transformations²⁹⁻³⁵.

Antimicrobial Activity: The synthesized complexes exhibited moderate antibacterial activity. While they did not surpass the efficacy of standard antimicrobial agents, their bioactive nature indicates potential for further development in antimicrobial applications²⁹⁻³⁵.

Biochemical Implications: The findings highlight the dual role of kynurenic acid as both a biologically active molecule and a precursor for metal complexes with promising catalytic and antimicrobial properties. This opens avenues for further research into the therapeutic applications of kynurenic acid and its metal complexes²⁹⁻³⁵.

In summary, the study demonstrates that kynurenic acid forms stable complexes with erbium, which possess excellent catalytic capabilities and moderate antibacterial activity. These findings underscore the potential of kynurenic acid as a versatile compound in both biological and chemical contexts, warranting further exploration of its applications in catalysis and antimicrobial therapy²⁹⁻³⁵.

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