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An Application of Catalytic and Antimicrobial Activity of Europium Rare Metal Complex with Quinoline Derivative

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Abstract: -

The combination of some rare metal ions with biologically important Quinoline derivative ligand to form coordination compound is an important area of current research. Less explored biologically important, Quinoline derivative ligand is allowed to react with solution of some rare metal perchlorates and attempt has been made to synthesize solid Quinoline derivative complexes. These Quinoline derivative complexes are subjected to U.V-Visible spectroscopy, IR spectroscopy, mass spectra, TGA analysis, elemental analysis etc. these complexes are used to study whether they possess catalytic activity in homogeneous or heterogeneous phase. Antimicrobial activity of these complexes has been evaluated by standard methods and attempts have been made to correlate structural characteristics with properties of these Quinoline derivative complexes.

Key Words:
Quinoline derivative, antimicrobial activity, antifungal activity, Europium Quinoline complex (Eu-KYNA).

1.0 Introduction

In the modern periodic table, two rows, are Ce-Lu and Th-Lr, are set apart from other elements. These two rows are collectively known as f block and are divided into lanthanides (Ce-Lu) and actinides (Th-Lr). All f block elements are metallic. There are in total fifteen lanthanide elements. They were discovered between 1794 (Y) and 1947 (Pm). The lanthanide ions have a +3 charge, but some elements have +2 and +4. [1-2]

2.0 Experimental

2.1Materials and Purification

Analytical grade chemicals were used throughout the course of experimental work. Spectroscopic grade solvents were employed for recording the spectra. Conductivity water was used throughout the work. Conductivity water was redistilled over alkaline potassium permanganate. The pH of this water was found to be ~ 6.9. This water was used for preparing solutions of metal perchlorates and reagents. Eu (III) perchlorate in DMSO solvent were prepared.

The compound kynurenic acid was used as a ligand. It was obtained from Sigma and its purity was checked by noting its melting point and spectra. All metal carbonates used were also A.R. grade.

2.2Preparation of complex

The formation of complex was carried out by mixing 50 ml 0.2M metal perchlorate in DMSO solution and 75 ml 0.2M ligand in DMSO solution. The mole ratio of ligand and metal was (1:1)

The reaction mixture was refluxed for 2.5 to 3.0 hours at 95 ^{0}C temperature. After 3.0 hours the reaction mixture was cooled. There was no immediate precipitation. The pH of the above solution was then raised up to 6.5 using 0.1M sodium hydroxide solution which resulted in the precipitation of the semi solid sticky material. Then, this solid product was dissolved in methanol to remove stickiness. The complex thus obtained was washed well with double distilled water to remove unreacted metal and ligand. All the complexes were

dried in oven at 40^O C to 50^{O}C.

2.3Analyses and Physical Measurements

M.P. and TLC [solvent system Toluene: Methanol (7:3)] were taken. TLC indicated single spot confirming presence of only complex species. Elemental analyses were performed with a Vario-MICRO CUBE C, H, N analyzer. There were two tubes (1) Combustion tube 1150 ^{0}C and (2) Reduction tube 850 ^{0}C. The gases used were helium and oxygen. The metal content was determined by titration with a solution of standardized disodium salt of EDTA after decomposing the complexes with a mixture of concentrated nitric acid, perchloric acid and sulfuric acid in 5:2:3 ml ratio, respectively [3-4]. Magnetic susceptibilities were measured by the Gouy's method [5-6] at room temperature, using $Hg[Co(CNS)_4]$ as calibrant. The IR spectra were recorded on a BRUKER ALPHA FT-IR 400 – 4000 cm^{-1} spectrophotometer. . The UV – visible spectra were measured on a UV-1800 Shimadzu (Double beam) spectrophotometer. Thermal measurements were performed using a METTLER TOLEDO STAR^e system TGA/DSC1(1150^{0}C) thermal analyzer. The mass spectra analyses were performed with a model QDA of Waters and Alliance 2690 analyzer.

3.0 Chemical kinetics

Three reactions (i)$K_2S_2O_8$ + KI (ii) $KBrO_3$ + KI and (iii) H_2O_2 + KI were selected. These reactions are usually carried out in neutral or acidic medium. The reactions are such that they proceed with moderate velocity K= 10^{-2} to 10^{-5} per minute. The product of all these three reactions is iodine which is titrated with standard aqueous sodium thio sulphate using starch solution as indicator. The rates of all these reactions can easily be measured by simple kinetic methods therefore one of the important applications of coordination compounds, as catalysts is being investigated. In present work, the setup of experiments and measurement of all the second order reactions has been carried out by standard procedures.[7-8] These reactions were carried out at room temperatures. Solutions of three complexes were prepared in methanol and in the blank sets, equal volume of methanol was added to equate the effect of solvent on the reaction. Catalytic amounts of complexes were added to the reaction systems. The experimental results are as follows:

Reactions: -

(i) Reaction-1

$K_2S_2O_8 + 2KI \longrightarrow 2K_2SO_4 + I_2$

$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$

(ii) Reaction-2

$KBrO_3 + HCl \longrightarrow KCl + HBrO_3$

$I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$

(iii) Reaction-3

$H_2O_2 + 2HI \longrightarrow 2H_2O + I_2$

$I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$

Table – 1 Reaction kinetics (without catalyst):

Reaction of : $K_2S_2O_8$ + KI + Methanol

Concentration : (0.0227M) (0.0227M) --

Volume : 50ml 50ml 10ml (t_∞ =113.5 ml)

Time t (min.)	Burette reading x (ml)	k = 1/at * x/(a-x) (lit.mol^{-1} min^{-1}
5	3.2	4.20 X 10^{-5}
10	3.7	2.44 X 10^{-5}
15	4.1	1.80 X 10^{-5}
20	4.6	1.52 X 10^{-5}
25	5.0	1.33 X 10^{-5}
30	5.5	1.22 X 10^{-5}

average k = 2.085 x 10^{-5}

a=b=initial concentrations of reactants =0.0227M

Table – 2 Reaction kinetics table without catalyst

Reaction of : $KBrO_3$ + KI + HCl + Methanol

Concentration : (0.0096M) (0.0096M) --

Volume : 25ml 25ml 10ml ($t\infty$ =25ml)

Time t (min.)	Burette reading x (ml)	k= 1/at * x/(a-x) (lit.mol^{-1} min^{-1}
5	6.9	3.04 X 10^{-3}
10	7.4	1.68 X 10^{-3}
15	7.7	1.18 X 10^{-3}
20	8.6	1.04 X 10^{-3}

25	**9.0**	**0.9 X 10^{-3}**
30	**9.5**	**0.81 X 10^{-3}**

average k = 1.44 X 10^{-3}

a=b=initial concentrations of reactants=0.0227M

Table – 3 Reaction kinetics table without catalyst

Reaction of : H_2O_2 + KI + H_2SO_4 + Methanol

Concentration : (0.0091M) (0.0091M) --

Volume : 10ml 10ml 10ml ($t\infty$ =50ml)

Time t (min.)	Burette reading x (ml)	k = 1/at * x/(a-x) ($lit.mol^{-1}$ min^{-1}
5	**1.2**	**9.8 X 10^{-5}**
10	**1.7**	**7.03 X 10^{-5}**
15	**2.3**	**6.42 X 10^{-5}**
20	**2.9**	**6.15 X 10^{-5}**
25	**3.4**	**5.83 X 10^{-5}**
30	**3.8**	**5.48 X 10^{-5}**

average k = 6.78 X 10^{-5}

a=b=initial concentrations of reactants =0.0227M

Table:- 4 Common Reaction Kinetics- experimental Set ups with Catalyst

Reactions (I)	**$K_2S_2O_8$ + KI + Eu-KYNA in 10 ml methanol (0.0227M) (0.0227M) (1 % MW)**	**$t\infty$ = 113.5 ml a=b= 0.0227M**
Reactions (II)	**$KBrO_3$ + HI + Eu-KYNA in 10 ml methanol (0.0091M) (0.0091M) (1 % MW)**	**$t\infty$ = 25 ml a=b= 0.0227M**
Reactions (III)	**H_2O_2 + HI + Eu-KYNA in 10 ml methanol (0.0091M) (0.0091M) (1 % MW)**	**$t\infty$ = 50 ml a=b= 0.0227M**

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Table:- 5 Kinetic experiments with Europium metal Complex

Reactions	k without Complexes	k with Eu –KYNA (1%)	% Increase reaction rate at T = 300K Eu -KYNA
$K_2S_2O_8$ + KI	2.085 X 10^{-5}	3.50X 10^{-5}	67.86 %
$KBrO_3$ + HI	1.44 X 10^{-3}	2.05 X 10^{-2}	1323.61 %
H_2O_2 + HI	6.78 X 10^{-5}	1.98 X 10^{-4}	192.03 %

k = reaction rate constant for the second order reaction, 1% complex = 1 % molecular weight of the complex

1 % MW of complex of Eu-KYNA= 0.0435 % of mole of $K_2S_2O_8$,

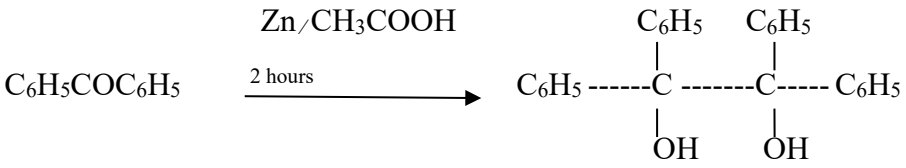
1 % MW of complex of Eu-KYNA = 0.104 % of mole of $KBrO_3$

1 % MW of complex of Eu-KYNA = 0.11 % of mole of H_2O_2

3.1 Catalysis of Organic Reaction

A mixture of benzophenone (7.5 gm, 0.041 mole) zinc dust (4 gm) glacial acetic acid (110 ml) and water (22 ml) is refluxed for 2 hours. [9-11] The solution is filtered (if necessary) and cooled. The separated benzpinacol is filtered and crystalline from glacial acetic acid. The yield was found to be 4.5 gm (30%).

The product melting point was 188-189 ^{0}C.[9-11]



Benzophenone **Benzpinacol**

Table:- 6 % yield of without catalyst for different temperature

Sr. No	Temperature	% yield without catalyst (for 4 hours reaction)	% yield without catalyst (for 3 hours reaction)	% yield without catalyst (for 2 hours reaction)
1	368 K	62.65%	52.45%	32.08 %

Table :-7 percentage yield with catalyst metal complexes for 2 hours

Temperature = 368 K

Complexes	For 1 % catalyst, yield obtained	For 5 % catalyst, yield obtained	For 10 % catalyst, yield obtained
Eu-KYNA	**29%**	**36%**	**58%**

1% MW of complex = 0.0243 % of mole of benzophenone
5% MW of complex = 0.121 % of mole of benzophenone
10% MW of complex = 0.243 % of mole of benzophenone

3.2 Results and Discussion

It was apparent that rates of all the redox reactions selected were increased by the addition of catalytic amounts of individual complexes. An increase of 68% was possible for reaction (i) $K_2S_2O_8$ + KI and for reactions (ii) $KBrO_3$ + KI + Hl and (iii) H_2O_2 + HI, a profound increase from 192% was possible. Thus a significant increase in reaction rates could be achieved with help of two complexes and hence application of these complexes as catalyst is certainly of immense significance.

The preparation of benzpinacol from benzophenone is an example of reductive coupling. The carbonyl group is reduced with zinc dust. Simultaneously, two units couple to form a new carbon-carbon bond in the center of the product molecule. Because this reaction is an example of two processes (reduction and new C-C bond formation) therefore it was chosen for possible application of Europium complexes as homogeneous catalysts. [12-16] The reaction was carried out with identical conditions for added catalysts and without catalyst. Eu-KYNA is acted as homogeneous catalyst for the above reaction. It was observed that addition of all the complexes in catalytic amounts drastically reduced the time requirement and increased the reaction yield. The highest increase was 58% and the lowest increase was 29%.Order of effectiveness as catalyst found was Eu-KYNA.

4.0 Antibacterial activity

4.1 Introduction: This part deals with the in-vitro screening of newly prepared compounds for antibacterial activity.[17-19] The species *S.aureus, E.coli, S.Pyogenes* and *P.Aeruginosa* [20-21]have been taken for the antibacteria11 activities. Agar-cup method was employed for the in-vitro screening for antibacterial activity. [22-23] The results of the Europium compound synthesized for antibacterial screening are mentioned in following Table.1

Table :- 8 Minimum Inhibitory Concentration (MIC) of Standard Antibacterial Drugs Against Selected Bacterial Strains

Standard Drug	E. coli *(MTCC 443)*	P. aeruginosa *(MTCC 1688)*	S. aureus *(MTCC 96)*	S. pyogenes *(MTCC 442)*	E. coli *(MTCC 443)*
Gentamycin	0.05 µg/mL	1 µg/mL	0.25 µg/mL	0.5 µg/mL	0.05 µg/mL
Ampicillin	100 µg/mL	—	250 µg/mL	100 µg/mL	100 µg/mL
Chloramphenicol	50 µg/mL	50 µg/mL	50 µg/mL	50 µg/mL	50 µg/mL
Ciprofloxacin	25 µg/mL	25 µg/mL	50 µg/mL	50 µg/mL	25 µg/mL
Norfloxacin	10 µg/mL	10 µg/mL	10 µg/mL	10 µg/mL	10 µg/mL

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

Table:- 9 Antibacterial activity of Quinoline derivative and its complex

Sr. No.	Compound Code	E. coli *(MTCC 443)*	P. aeruginosa *(MTCC 1688)*	S. aureus *(MTCC 96)*	S. pyogenes *(MTCC 442)*
1	**KYNA ligand**	100 µg/mL	250 µg/mL	250 µg/mL	200 µg/mL
2	**Eu-KYNA**	85 µg/mL	205 µg/mL	237 µg/mL	187 µg/mL

The synthesized quinoline derivative (KYNA ligand) and its osmium complex (Eu-KYNA) exhibited **moderate antibacterial activity** against all tested bacterial strains. The osmium complex (Eu-KYNA) demonstrated slightly better activity than the free ligand, suggesting a possible enhancement of biological activity upon complexation. However, both compounds showed **higher MIC values (lower potency)** compared to the standard antibiotics.

Comparison of the antibacterial activity of the synthesized complexes with standard antibacterial agents revealed that, although the complexes exhibited moderate to good activity against all four bacterial strains, their activity was consistently lower than that of the standard antibiotics [24-26].

2.0Antifungal 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*.[27-30] 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 10.

Table:-10 Minimum Inhibitory Concentration (MIC) of Standard Antifungal Drugs Against Selected Fungal Strains

Standard Drug	C. albicans *(MTCC 227)*	A. niger *(MTCC 282)*	A. clavatus *(MTCC 1323)*
Nystatin	100 µg/mL	100 µg/mL	100 µg/mL
Griseofulvin	500 µg/mL	100 µg/mL	100 µg/mL

Table:- 11 Minimum Fungicidal Concentration (MFC) of Synthesized Compounds Against Selected Fungal Strains

Sr. No.	Compound Code	C. albicans *(MTCC 227)*	A. niger *(MTCC 282)*	A. clavatus *(MTCC 1323)*
1	**KYNA ligand**	1000 µg/mL	500 µg/mL	500 µg/mL
2	**Eu-KYNA**	567 µg/mL	998 µg/mL	1025 µg/mL

The comparative analysis of the antifungal activity indicates that, although the synthesized complexes demonstrated reasonable to significant inhibitory effects against the tested fungal strains,[31-36] their effectiveness did not surpass that of the standard antifungal agents used for reference.

4.4 Result and discussion: -

Results of antibacterial activities of the complexes recommended that Eu-KYNA complex exhibited equal activity as standard drug Ampicillin towards E.coli. Against S.aureus, Eu-KYNA showed equal activity and greater activity was exhibited by complexes paralleled to standard Ampicillin drug. The outstanding antibiotics exhibited greater activities compared to the antibacterial performance of the complexes. The antifungal activities of all the complexes were found to be less than that of standard antifungal antibiotic drugs.

In the complexes showed some capable results against selected bacteria and they, along with others, may be further explored against other organisms too. There are some chances of getting encouraging outcomes.

4.5 Conclusion

Quinoline derivatives are key biologically active molecules with significant physiological functions. To better understand their biological roles, complexation behavior, and biochemical properties, osmium complexes of a quinoline derivative were synthesized in the present study. These complexes were characterized structurally and evaluated for their catalytic and biological activities. The results were encouraging, as the quinoline derivative demonstrated a strong tendency for complex formation and exhibited promising catalytic properties, along with moderate antibacterial activity. Quinoline-based complexes have been widely reported as effective catalysts,

particularly in enhancing the reaction rates of selected organic transformations involving redox processes and C–C bond formation.

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