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Research Article

Synthesis, Characterization And Anti-Microbial Activity Of C- 3 Substituted Lawsonemonoximates Of Ytterbium (Iii)

Shrikant B. Jagtap

Abstract

An organic compound with presence of hetero atoms shows very interesting characteristics. They show a wide range of application in the field of pharmaceutical, analytical and co-ordination chemistry. They also possess a variety of biological application. When these compounds are used in co-ordination chemistry and chelated with different metals gives rise to various physio chemical properties. They also tend to show a very good biological activity. In present work C-3 substituted Lawsonemonoximates are synthesized and chelates of Yetterbium (III) are prepared. The antimicrobial activity of the compounds was determined by disk diffusion method and broth micro-dilution techniques using Mueller Hinton medium against the following organisms.

Keywords: C- 3 Substituted Lawsonemonoximate, oximes, Ytterbium, antimicrobial activity.

1. Introduction

Lawsonemonoximates is a very interesting organic compound which exhibits various biological properties. The utilization of the lanthanides and their complexes in biological and biochemical studies has been reviewed by Williams[l]. The application for localization of tumors using appropriate lanthanide complexes was studied by Hider et al.[2] and Lauffer[3]. Yam et al. have reported that lanthanide complexes play a key role in various diagnostic areas[4]. Gaikwad[5] and Dandawate[6] have studied the complexes of some lanthanide metals with hydroxynaphthoquinone-oximes.

Lawsone and its dertive have a great potential in the various fields of chemistry. Aspects of inorganic, coordination chemistry and biological activity are explored in the further work. But these compound and its chelates possess a larger scope for research.

Among the finest route to increase the antitumor activity and reduce the side effects of metal complexes is their abstract into starch, lipid and liposome microspheres. Nanocapsules provides a huge range of exciting features across their micro-sized correspondents as they hold more surface area, have increased solubility, significantly higher biocompatibility, biodegradability, stability in the course of storage, and controlled release.[7,8]

[1] Coordinator, Research Centre in Chemistry, Annasaheb Magar Mahavidyalaya, Hadapsar
drshrikantjagtap@yahoo.com

As an extension of our previous paper,[9-14] we decided to reveal the luminescence of the Yb(III) containing Me_2Phen ligand, $[Yb(Me_2Phen)_2Cl_3(OH_2)]$ and establish it as a novel probe to BSA (Bovine Serum Albumin) and FS-DNA (Fish-Salmon DNA). Thus, the binding of the ytterbium complex with BSA and FS-DNA was investigated by emission spectroscopy, UV-vis titration, viscosity measurement, CD spectroscopy, and docking method. Also, the ability of this complex to cleave DNA by gel electrophoresis was declared. Moreover, nanocarriers of this complex produced, and the anticancer, antifungal, and antibacterial properties of this complex studied.

2. Materials and Methods

2.1 Preparation of Ligand:

All the chemicals and solvents used were of A.R grade. 2-hydroxy-l,4-naphthalenedione(lawsone), 2,3-dichloro-l,4-naphthalenedione(dichlone) and 2-methyl-l,4-naphthalenedione (menadione) these organic chemicals were purchased from Fluka (Germany). 2-hydroxy-3-methyl-l, 4-naphthalenedione (phthiocol) was prepared from menadione by Fieser's method [15]. The 2-hydroxy-3-chloro-l, 4-naphthalenedione was synthesized from dichlone. All the ligands (2-hydroxy-l, 4-naphthalenedione-l-oxime derivatives) were prepared by the method reported in earlier paper [16].They were recrystallized using methanol and melting points were recorded.

2.2 Preparation of Chelates:

3 mmol of oxime derivative [0.568 g of (I), 0.60 g of (II), 0.671 g of (III), 0.804 g of (IV) and 0.945 g of (V)] solution was heated and added in 25 mL of ethanol, an aqueous solution of mmol of ytterbium trichloride hexahydrate (0.381 g) was added with constant stirring. The pH of the solution mixture was maintained in between 5-7 by adding ammonia. It was refluxed for 3 h and then cooled overnight. The precipitate was filtered off, washed with water, followed by hot methanol[17] and dried in vacuum over fused $CaCl_2$ at ambient temperature.

2.3 Anti-microbial activity of the compound:

Media: The dehydrated plate count medium (g/100 ml distilled water glucose 0.1, yeast extract 0.25, tryptone 0.5) and Sabouraud's dextrose agar g/100 ml distilled water glucose 4, peptone) purchased from Hi- Media Laboratories, India were used respectively for antibacterial and antifungal activity.

Maintenance of Culture: The stock cultures of these microorganisms were maintained at -20C in 15% glycerol. The inoculum was prepared from stock cultures by streaking onto the plate count agar for bacteria and on Sabouraud's dextrose agar for fungi. After an overnight incubation single colony was used to inoculate sterile liquid media. The 5ml broth was dispensed in test tubes and sterilized in the autoclave at 121C for 15 min. The broths were then inoculated with respective cultures and incubated on an orbital shaker (150 rpm) overnight at 30ºC A540 of bacterial cultures. Zone of inhibition was determined by the Well diffusion assay method.

2.4 Methods for Magnetic Moment and Elemental Analysis:

The magnetic studies were carried out at room temperature by the Faraday technique using Mercury (II) tetrathiocyanatocobaltate as calibrant. The elemental analysis was carried out using a Hosli-Holland C, H Analyzer.

3. Results and Discussion

The prepared compounds were characterized by using various techniques such as magnetic moment, elemental analysis, UV-VIS, IR, ^{1}H NMR and ^{13}C NMR.

3.1 Analytical Data and Magnetic Moment of Yb (III):

Complex	% Yield	Elemental Analysis				μeff/B.M.
		C/%	H/%	N/%	M/%	
Yb1	74.12	46.12	2.57	5.18	22.54	4.13
Yb2	73.89	48.41	3.49	5.02	21.25	4.40
Yb3	70.52	40.82	2.36	4.59	19.50	4.29
Yb4	72.03	36.83	2.07	4.23	16.90	4.36
Yb5	66.14	31.24	1.91	3.50	15.39	4.21

The elemental analysis of ligands is already reported [18]

3.2 IR Analysis of the compounds:

IR analysis of these compounds shows the presence of various functional groups present in the compounds. Following table shows the IR frequencies obtained for the organic compound.

Compound	υ (O-H)	υ (C=O)	υ (C=N)	υ (N-O)
Yb 1	3362,3155 3267	1630- 1588	1576 1538	1059
Yb 2	3275, ’3100 3314	1620 1586	1587 1526	1060
Yb 3	3412,3375, 3100 3567	1604 1580	1577 1521	1059
Yb 4	3325,3200, 3100 3184	1623 1580	1589 1518	1060
Yb 5	3325,3187, 3100 3440	1620 1579	1585 1521	1055

3.3 ^{1}H NMR Study of the Compounds:

^{l}H NMR data for (I)-(V) is depicted in Table 3and 4 respectively. The syn-amphi isomers of 2-hydroxy-l,4-naphthalenedione -l- oxime derivatives are shown in the following table:

^{1}H NMR data for lawsone and its oxime derivatives (ppm)

Compound*	N-OH	C_2-OH	H_3	H_5	$H_{6,7}$	H_8 - CH_3	H_8 - CH_3
Lawsone	-	11.66(br)	6.15(s)	7.90(d)	7.79(q)	7.95(d)	
(I)	9.05(d)	-	6.16(s)	8.00(d)	7.63(q)	8.22(d)	
(II)	9.01(d)	12.72	-	8.15(d)	7.57(q)	8.29(d)	2.05
(III)	9.02(d)	-	-	8.15(d)	7.61(q)	8.30(d)	
(IV)	9.06(d)	13.61	-	8.16(d)	7.66(q)	8.27(d)	
(V)	9.04(d)	13.44	-	8.16(d)	7.61(q)	8.28(d)	

*Ligand, br-broad, s-singlet, d- doublet, q- quartet

The C2-OH signal for (II), (IV) and (V) appeared in the region 12.72-13.61 ppm is indicative of its participation in intramolecular hydrogen bonding. However, the spectrum for (I) and (III) do not exhibit any signal originating from this C2-hydroxyl group probably representing the increased stability of intramolecular hydrogen bonding.

3.4 Antimicrobial Activity of the compound and its chelates with Yb

Anti-microbial activities of the complexes were tested against various bacteria such as S. aures, X. campestris, P. aeruginosa, C. albicans etc. Zone of inhibition of the respected the compounds were measured and are given in the following table

Compound	S. aures	X. campestris	P. aeruginosa	C. albicans	A. niger
Dichalcone	15	8	9	16	8
Yb_1	11	9	9	11	12
Yb_2	17	11	10	15	19
Yb_3	12	10	8	15	18
Yb_4	8	8	8	9	15
Yb_5	10	8	8	12	15

The extent of inhibition of microorganisms by ligands and its complexes is in the following order.

Microorganisms	Compounds
S. aures	L2>L1>Yb2>dichlone,L3>L5>L4>Yb3>Yb1>Yb5>Yb4
X. campestris	L2>L3>L1,L5>Yb2>L4>Yb3>Yb1> dichlcone>Yb4>Yb5
P. aeruginosa	L2>L1>L3>Yb2>dichlcone>L4>L5>Yb1>Yb3,Yb4>Yb5
C. albicans	L2, L3>L4, L5>L1>dichlcone>Yb2>Yb3>Yb5>Yb1>Yb4
A. niger	L3>L3, L4, L5>Yb2, Yb3, Yb4, Yb5>L1>Yb1>dichlone

Decrease in the activity may be due to partial dissociation of chelate. Thus releasing small amount of active component at the site of action in the cell. Hence, metal enzyme activity essential for survival of micro-organism may not be affected.

4. Conclusion

The interaction of the ligands (table 1) with metal salts in 1:3 molar ratio in ethanol yielded stable solid complexes analogous to the molecular composition supported by elemental analysis is $YbL_3(H_20)_2$ (where L anion of the corresponding ligands).Spectroscopic data i.e. IR and ^1H NMR gives the validity of the synthesised compound. Anti-microbial activities of the complexes were tested against various bacteria such as S. aures, X. campestris, P. aeruginosa, C. albicans etc. When the organic compounds are tested for the anti-microbial activity they show higher activity than the chelated complexes with Ytterbium.

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