ORIGINAL ARTICLE

Synthesis, Characterization, Catalytic and Antimicrobial Activities of Benzylidene Imines with Ruthenium (III) Complexes

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ABSTRACT

The synthesis and characterization of several hexa – coordinated Ru(III) complexes of the type $[RuCl(CO)(EPh_3)_2(L)]$ (E = P or As ; L = monobasic bidentate anion) are reported. IR, electronic, EPR, electrochemistry and catalytic activity of the complexes are discussed. An octahedral geometry has been tentatively proposed for all the synthesized complexes. These complexes catalyze oxidation of alcohols into their corresponding aldehydes or ketones. The new complexes have been subjected to antimicrobial investigation also.

Keywords: Ruthenium(III) Complexes, Synthesis, Characterization, Catalytic oxidation, Antimicrobialactivity.

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INTRODUCTION

In the recent years there has been considerable interest in the chemistry of transition metal complexes of Schiff bases [1]. The metal ruthenium possesses several favorable chemical properties that indicate it may be a strong candidate to form a basis for rational anticancer drug design [2-3]. Benzylidene imines have vast medicinal importance showing a number of activities such as tuberculostatic, bactericidal, fungicidal, anti-inflammatory etc.,[4-6]. The pharmacological importance of imidazolones and imines are also reported [7-10]. Bidentate complexes have been employed as catalyst for many reactions and as biological models in understanding the structure of bio-molecules and biological process [11 -12]. They are increasingly important for designing metal complexes related to synthetic and natural oxygen carriers [13].

The real impetus towards developing their coordination chemistry was their physicochemical properties and significant biological activities [14 -15]. The chemistry of ruthenium is currently receiving a lot of attention, primarily because of the fascinating electron transfer properties displayed by the complexes of this metal [16]. Ruthenium offers a wide range of oxidation states and the reactivity of the ruthenium complexes depend on the stability and interconvertibility of these oxidation states, which in turn depend on the nature of the ligand bound to the metal complexation of ruthenium by ligands of different types has thus been of particular interest [17-18]. Herein, we are reporting the preparation, spectral, catalytic activity and antimicrobial activities of some Ru(III) complexes containing bidentate ligands. The general structure of the ligands are given in Scheme 1.

MATERIAL AND METHODS

All the reagent used were of analar or chemically pure grade. Solvents were purified and dried according to standard procedures. RuCl₃.3H₂O purchased from Loba Chemie was used without further purification. The carbon, hydrogen and nitrogen analyses were performed at the Central Drug Research Institute in 2019, Lucknow, India. IR spectra of the complexes were recorded as KBr pellets on a shimadzu 8000 FT-

IR spectrophotometer in the 4000 - 400 cm⁻¹ range at PSGR Krishnammal college for women in 2019 at Coimbatore. The electronic spectra were recorded in CH_2Cl_2 solution with a systronics 119 spectrometer in the 800 – 200 nm range at P.S.G.R. Krishnammal College for Women in 2019 at Coimbatore. EPR spectra of the powdered samples were recorded Ea Bruker on a E-112 Varian model instrument in X-band frequencies at room temperature at Indian Institute of science in 2019 at Bangalore. Magnetic susceptibilities were recorded on EG and G-PARC vibrating magnetometer. The cyclic voltametric studies were carried out with BAS CV-27 model electrochemical analyzer in acetonitrile solution using a glassy carbon working electrode and potentials were referenced to saturated calomel electrode. Catalytic activity of metal complexes were studied in 2019 at Biotechnology Department, Bharathiar University, Coimbatore. Melting points were recorded with a Raaga heating table and are uncorrected.

Procedure

The starting complexes $[RuCl_3(PPh_3)_3]$ [19], $[RuCl_3(AsPh_3)_3]$ [20], $[RuBr_3(AsPh_3)_3]$ [21] and $[RuBr_3(PPh_3)_2(MeOH)]$ [22] and the ligands [23] were prepared according to the literature procedures. Catalytic oxidation of alcohols and antibacterial studies were carried out according to our earlier procedure [24-25].

Preparation of new ruthenium (III) complexes

All the reactions were carried out under anhydrous condition. To a solution of $[RuX_3(EPh_3)_3]$ (E = P or As , X = Cl or Br) (0.1g, 0.08 - 0.01 mmol) or $[RuBr_3(PPh_3)_2(MeOH)]$ (1.12 g, 0.01 mmol) in benzene (20ml) the respective ligands (0.03-0.08 g, 0.08 - 0.1 mmol) were added. The resulting solution was concentrated and the product was separated by the addition of the small amount of pet. ether (60 - 80 °C). It was filtered and recrystallized in $CH_2Cl_2/light$ pet. ether (60 - 180 °C) and dried in vacuum (yield = 70 - 85%).



Scheme 1.Structure of the ligand

R ₁	R ₂	R ₃	Abbreviation
Н	CH ₃	Н	HL_1
OCH ₃	CH ₃	OCH ₃	HL ₂
ОН	CH ₃	OH	HL_3
ОН	CH ₃	Н	HL_4

RESULTS AND DISCUSSION

Light and air stable ruthenium(III) complexes of the general formula $[RuX(EPh_3)_2(L)(CO] (E = P \text{ or } As; L = monobasic bidentate anion)$ have been prepared by reacting $[RuX_3(EPh_3)_3]$ and $[RuBr_3(PPh_3)(MeOH)]$ with the respective ligands in an 1:1 molar ratio in benzene (Scheme 2).



Scheme 2. Synthesis of Ru(III) Complexes

The analytical data obtained for the new complexes (Table 1) are agree very well with the proposed molecular formulae in all of the above reactions, the ligand behave as monobasic bidentate ligands. *IR spectra*

The IR spectra of the free ligands were compared with those of the new complexes in order to confirm the coordination mode of benzylidene imine to the ruthenium metal (Fig 1& 2). The IR spectrum of the free ligands showed a band in the absorption due to (C=N) appears in the 1673 - 1630 cm⁻¹ region undergoes a negative shift by 5 - 35 cm⁻¹ in the spectra of the complexes indicating the co-ordination of azomethine to the metal [26]. A strong band which appeared in the spectra of the ligands around 1181 cm⁻¹ due to v (C = 0) completely disappeared and a new band was observed around 1093 cm⁻¹. This may be due to the enolisation and subsequent coordination through the deprotonated oxygen atom of the CH–C=O group [27]. In addition all the complexes show a new band at 1520 cm⁻¹ due to the C=O group (Table 2). This

band remains unaltered in the corresponding metal complexes indicating the non-involvement of this group on complexation [28]. In addition to the above, the characteristic bands due to PPh_3 or $AsPh_3$ were also present in the expected region [29-30].

Electronic spectra

The electronic spectra showed two to three bands in the 290-690 nm region (Fig 3 & 4). The ground state of ruthenium(III) is ${}^{2}T_{2g}$ and the first excited doublet levels in order to increasing energy are ${}^{2}A_{2g}$ and ${}^{2}T_{1g}$ which arises from $t_{52g} e_{1g}$ configuration [31]. In the most of the ruthenium(III) complexes the electronic spectra showed only charge transfer bands [32]. The band in the 690 – 595 nm region have been assigned to the d–d transition. Which is in conformity with assignments made for the similar ruthenium(III) complexes [33 - 34]. Other bands in the 375 – 230 nm region have been assigned to the charge transfer transitions [35]. In general the electronic spectra of all the complexes are characteristic of an octahedral environment around ruthenium(III) ions.

EPR spectra

All the ruthenium(III) complexes are paramagnetic showing a +3 oxidation state for ruthenium ion. The solid state EPR spectra at X-band frequencies for several new ruthenium(III) complexes have been recorded at room temperature(Fig 5 & 6). The g value of the complexes are listed in Table 3. All the complexes shows a single isotropic resonances with a "g" value in the range. The isotropic lines of the type usually observed are either due to the intermolecular spin exchange, which may be broaden the lines or to the occupancy of the unpaired electron in degenerate orbital. The nature and pattern of the EPR spectrum suggests an almost perfect octahedral environment around ruthenium ion in these complexes [36-37].

Magnetic moments

The magnetic moments for ruthenium(III) complexes have been measured at room temperature using a vibrating sample magnetometer and diamagnetic corrections have been applied. The value obtained were from 1.90 - 2.00 BM indicating the presences of one unpaired electron, suggesting a low spin t_2g^5 configuration for the ruthenium(III) ion an octahedral environment in all of these complexes [38].

Electrochemistry

Complexes were electrochemically examined at a glassy carbon working electrode in dichloromethane solution using cyclic voltammetry. The potential data are listed in Table 4. The complexes display the Ru(III) – Ru(II) and Ru(III)–Ru(IV) couples in the potential ranges –0.43 to –0.67 and 0.83 to 1.17 V respectively vs. SCE. In this, the Ru(III)–Ru(II) redox couple is quasi-reversible in nature, with a peak-to-peak separation (Δ Ep) of 120–370 mV, and the Ru(III)–Ru(IV) couple is irreversible. The reason for the irreversibility of these complexes may be oxidative degradation or the short-lived oxidized state of the metal ion [38-40].

Catalytic activity



R, R' = alkyl (or) aryl

Scheme 3. Catalytic oxidation of alcohols

The oxidation of alcohols was carried out with the ruthenium complexes as catalyst in the presence of *N*-methylmorpholine –*N*-oxide (NMO) as co-oxidant in chloroform (Scheme 3). Benzaldehyde was formed from benzylalcohol and cyclohexanol was converted into cyclohexanone after stirring for 3 h at room temperature. The products formed were quantified as their 2, 4-dinitrophenyl hydrazone derivatives. In no case, there was any detectable oxidation of alcohols in the presence of NMO alone and without ruthenium complexes. All of the synthesized ruthenium complexes were found to catalyze the oxidation of alcohols to carbonyl compound but the yield and turnover were found to vary with different catalyst [Table 5]. The yield and turnover number are comparable with those reported for the oxidation of alcohols by similar ruthenium(III) complexes [41]. It has also been found that PPh₃ complexes possess higher catalytic activity than the AsPh₃ complexes [42]. This may be due to the higher donor ability of the arsine ligand compared to the phosphine ligand. The relative higher product yield obtained for the oxidation of benzyl alcohol than for cyclohexanol is due to the fact that the - CH moiety of benzyl alcohol

is more acidic compared to that of cyclohexanol [43-44]. The catalytic oxidation is expected to proceed *via* Ru(IV)=0 intermediate as reported by us earlier [45].

Antimicrobical study

The *in vitro* antimicrobial screening of the ligands and their ruthenium complexes have been carried out against Eschericha coli, Aeromonas hydrophila and Salmonella typhi using a nutrient agar medium by disc diffusion method [46]. The results (Table 6) showed the complexes exhibit moderate activity against Eschericha coli, Aeromonas hydrophila and Salmonella typhi is shown by Fig 7. The toxicity of ruthenium chelates increases on increasing the concentration [47]. The increase in the antimicrobial activity of the metal chelates may be due to the effect of the metal ion on the normal cell process. A possible mode of the toxicity increase may be considered in light of Tweedys chelation theory [48]. Chelation considerable reduces the polarity of the metal ion because of partial sharing of its positive charge with the donor groups and possible π - electron delocalization over the whole chelate ring. Such chelation could enhance the lipophilic character of central metal atom, which subsequently favors its permeation through the lipid layers of cell membrane. Furthermore, the mode of action of the compounds may involve formation of a hydrogen bond through the azomethine (>C=N) group with the active centers of cell constituents, resulting in interference the normal cell process [49]. Though the complexes possess activity they could not reach the effectiveness of the standard drug streptomycin. The variation in the effectiveness of the different compounds against different organism depend either of impermeability of the cells or the microbe of difference in ribosome of microbial cells [50].

Based on the analytical and spectral data of IR and absorption, an octahedral geometry (Scheme 4) has been tentatively proposed for all the synthesized ruthenium(III) complexes.



Scheme 4. Structure of new Ru(III) complexes
$(R_1 = H, OCH_3, OH; R_2 = CH_3; R_3 = H, OCH_3, OH; E = P or As)$

Complex	Mp (°C)	Yield (%)	Found (Calculated) (%)		
			С	Н	Ν
$[RuBr(CO)(PPh_3)_2(L_1)]$	168	75	61.68	4.24	4.68
			(61.69)	(4.22)	(4.64)
[RuCl(CO)(AsPh ₃) ₂ (L ₁)]	171	85	61.76	4.23	4.64
			(61.79)	(4.23)	(4.65)
[RuCl(CO)(PPh ₃) ₂ (L ₂)]	179	74	65.31	4.67	4.74
			(65.31)	(4.67)	(4.75)
[RuBr(CO)(PPh ₃) ₂ (L ₂)]	168	72	60.92	4.35	4.43
			(60.95)	(4.36)	(4.44)
$[RuCl(CO)(AsPh_3)_2(L_2)]$	173	81	60.75	4.32	4.48
			(60.75)	(4.35)	(4.43)
$[RuCl(CO)(PPh_3)_2(L_3)]$	170	83	64.79	4.45	4.86
			(64.80)	(4.44)	(4.87)
[RuBr(CO)(PPh ₃) ₂ (L ₃)]	165	80	60.08	4.12	4.51
			(60.09)	(4.12)	(4.52)
$[RuCl(CO)(AsPh_3)_2(L_3)]$	168	77	60.20	4.11	4.53
			(60.21)	(4.13)	(4.53)
[RuCl(CO)(PPh ₃) ₂ (L ₄)]	166	79	65.72	4.51	4.93
			(65.72)	(4.51)	(4.93)
$[RuBr(CO)(PPh_3)_2(L_4)]$	175	80	60.87 4.16 4		4.56
			(60.87)	(4.16)	(4.58)
$[RuCl(CO)(AsPh_3)_2(L_4)]$	179	82	60.97	4.18	4.58
			(60.98)	(4.18)	(4.58)

Table 1.Analytical data of new Ru(III) Complexes

Tuble, 2. In and electronic spectral data of the new ruthemum(in) complexes										
S.NO	Complex	^v (C=N) cm ⁻¹	^υ (C=0) cm ⁻¹	PPh ₃ /AsPh ₃	λ _{max} nm					
	H-L ₁	1673	1181							
	H-L ₂	1625	1268							
	H-L ₃	1610	1270							
	H-L ₄	1630	1267							
1	$[RuCl(CO)(PPh_3)_2(L_1)]$	1638	1093	1434,1085	690,400,250					
2	$[RuBr(CO)(PPh_3)_2(L_1)]$	1595	1560	1434,1085	690,390,250					
3	$[RuCl(CO)(AsPh_3)_2(L_1)]$	1600	1565	692	680,360, 280					
4	$[RuCl(CO)(PPh_3)_2(L_2)]$	1605	1570	1433,1086,694	670,370, 260					
5	$[RuBr(CO)(PPh_3)_2(L_2)]$	1590	1565	1436,1078, 670	680,350, 270					
6	$[RuCl(CO)(AsPh_3)_2(L_2)]$	1600	1560	1437, 1093, 682	690,350, 280					
7	$[RuCl(CO)(PPh_3)_2(L_3)]$	1595	1565	1435, 1086, 696	680,370, 270					
8	$[RuBr(CO)(PPh_3)_2(L_3)]$	1605	1565	1433, 1084, 692	680,370, 260					
9	$[RuCl(CO)(AsPh_3)_2(L_3)]$	1595	1570	1432, 1078, 694	690,350, 260					
10	$[RuCl(CO)(PPh_3)_2(L_4)]$	1585	1570	1431, 1092, 670	680, 280					
11	$[RuCl(CO)(AsPh_3)_2(L_4)]$	1600	1560	1436, 1076, 684	690,350, 270					
12	$[RuBr(CO)(PPh_3)_2(L_4)]$	1585	1570	1434, 1074, 690	680,370, 290					

Table. 2. IR and electronic spectral data of the new ruthenium(III) Complexes

Table 3. EPR data and magnetic moment of Ruthenium(III) complexes

Complex	gx	gу	gz	g*	μm
$[RuCl(CO)(PPh_3)_2(L_1)]$	2.15	2.15	2.15	2.15	1.92
$[RuBr(CO)(PPh_3)_2(L_1)]$	2.17	2.17	2.17	2.17	n
$[RuCl(CO)(AsPh_3)_2(L_1]$	2.19	2.19	2.19	2.19	1.90
$[RuCl(CO)(PPh_3)_2(L_2)]$	2.3	2.3	2.3	2.3	n
$[RuBr(CO)(PPh_3)_2(L_2)]$	2.28	2.28	2.28	2.28	1.96
$[RuCl(CO)(AsPh_3)_2(L_2)]$	2.35	2.35	2.35	2.35	1.93
$[RuCl(CO)(PPh_3)_2(L_3)]$	2.32	2.32	2.32	2.32	1.97
$[RuBr(CO)(PPh_3)_2L_3]$	2.18	2.18	2.18	2.18	n
$[RuCl(CO)(AsPh_3)_2(L_3)]$	2.20	2.20	2.20	2.20	2.00
$[RuCl(CO)(PPh_3)_2(L_4)]$	2.27	2.27	2.27	2.27	n
[RuCl(CO)(PPh ₃) ₂ (L ₄)]	2.25	2.25	2.25	2.25	n
$[RuCl(CO)(AsPh_3)_2(L_4)]$	2.22	2.22	2.22	2.22	1.98

 $g^* = (1/3g_x^2 + 1/3g_y^2 + 1/3g_z^2)$ n = not recorded

Table 4. Cyclic Voltammetry data for some Ruthenium(III) complexes

Compleyes	Ru(iv) -Ru(iii)				Ru(iii)-Ru(ii)			
complexes	Epa(V)	Epc(V)	Ef(V)	ΔEp(mV)	Epa(V)	Epc(V)	Ef(V)	ΔEp(mV)
[RuCl(CO)(PPh ₃) ₂ (L ₁)]	-	-	-	-	-0.05	-0.2	-0.075	250
[RuCl(CO)(PPh ₃)(L ₂)]	0.06	0.45	0.525	150	-0.3	-0.25	-0.125	250
[RuCl(CO)(PPh ₃) ₂ (L ₃)]	-	-	-	-	-0.05	-0.3	-0.175	250
[RuCl(CO)(PPh ₃) ₂ (L ₄)]	0.05	0.4	0.225	350	-0.825	-0.66	-0.782	165
$[RuCl(CO)(PPh_3)_2(L_5)]$	-	-	-	-	-0.2	-0.55	-0.325	350

Complex	Substrate	Product	Yield ^b	Turnover ^c
$[RuCl(CO)(PPh_3)_2(L_1)]$	Benzylalcohol	А	71	75
	cyclohexanol	К	63	67
$[RuBr(CO)(PPh_3)_2(L_1)]$	Benzylalcohol cyclohexanol	А	67	71
		К	56	60
$[RuCl(CO)(AsPh_3)_2(L_1)]$	Benzylalcohol cyclohexanol	А	82	86
		К	75	79
$[RuCl(CO)(PPh_3)_2(L_2)]$	Benzylalcohol cyclohexanol	А	75	76
		К	65	69
$[RuBr(CO)(PPh_3)_2(L_2)]$	Benzylalcohol cyclohexanol	А	80	84
		К	73	77
$[RuCl(CO)(AsPh_3)_2(L_2)]$	Benzylalcohol cyclohexanol	А	70	74
		К	62	66
$[RuCl(CO)(PPh_3)_2(L_3)]$	Benzylalcohol cyclohexanol	А	75	79
		К	66	70
$[RuBr(CO)(PPh_3)_2(L_3)]$	Benzylalcohol cyclohexanol	А	73	77
		К	59	63
$[RuCl(CO)(AsPh_3)_2(L_3)]$	Benzylalcohol cyclohexanol	А	72	76
		К	65	65
$[RuCl(CO)(PPh_3)_2(L_4)]$	Benzylalcohol cyclohexanol	А	72	76
		К	65	65
$RuBr(CO)(PPh_3)_2(L_4)]$	Benzylalcohol cyclohexanol	А	70	74
		К	63	68
$[RuCl(CO)(AsPh_3)_2(L_4)]$	Benzylalcohol cyclohexanol	А	80	84
		К	74	78

Table 5.Catalytic oxidation of alcohols by new Ru(III) complexes in the presence of NMO

^aA: Benzaldehyde ; K: Cyclohexanone

^b Yield based on susbstrate
^c Moles of product per more of catalyst

Table 6.Antimicrobial activity of ligands and Ru(III) new complexes

Ligand/Complex	Diameter of inhibition Zones (mm)								
	Escherichia coli Aeromonas hychophla			Salmo	<u>Salmonella typhi</u>				
Ligand/Complex %	0.25%	0.5%	1%	0.25%	0.5%	1%	0.25%	0.5%	1%
HL ₁	10	12	13	9	10	12	8	10	11
$[RuCl(CO)(PPh_3)_2(L_1)]$	12	14	16	11	14	17	10	12	15
$[RuBr(CO)(PPh_3)_2(L_1)]$	11	13	15	10	11	13	10	12	14
$[RuCl(CO)(AsPh_3)_2(L_1)]$	12	13	14	11	12	12	9	14	15
HL ₂	10	11	13	10	12	13	10	12	13
$[RuCl(CO)(PPh_3)_2(L_2)]$	12	14	17	12	14	19	12	14	15
$[RuBr(CO)(PPh_3)_2(L_2)]$	11	13	16	11	13	15	11	13	14
$[RuCl(CO)(AsPh_3)_2(L_2)]$	12	13	14	12	13	14	11	13	15
HL ₃	10	11	12	9	10	11	9	11	12
$[RuCl(CO)(PPh_3)_2(L_3)]$	13	14	16	10	12	14	10	12	15
$[RuBr(CO)(PPh_3)_2(L_3)]$	12	15	16	10	11	13	10	12	14
$[RuCl(CO)(AsPh_3)_2(L_3)]$	12	13	12	10	11	12	13	14	15
HL_4	9	10	12	8	10	11	8	11	12
$[RuCl(CO)(PPh_3)_2(L_4)]$	11	13	14	10	12	13	11	12	13
$[RuBr(CO)(PPh_3)_2(L_4)]$	11	12	13	10	11	13	10	11	14
$[RuCl(CO)(AsPh_3)_2(L_4)]$	10	11	13	10	11	12	11	12	13
Streptomycin	22	23	28	21	27	29	29	21	25











Fig: 5 EPR Spectrum of [RuCl(CO)(PPh₃)₂(L₁)]



Fig: 6 EPR Spectrum of [RuCl(CO)(AsPh₃)₂(L₁)]





Fig: 7 Antimicrobial activity of [RuCl(CO)(PPh₃)₂(L₁)]complexes

SUMMARY AND CONCLUSION

The present work deals with the synthesis and characterization of ruthenium complexes. The structures of the complexes were determined by analytical and spectral studies. The complexes were tested for their antimicrobial activity.

The following conclusions were derived from the present work:

- The synthesized Schiff bases act as mono basic bidentate ligands.
- The studies show octahedral geometry for Ru(III) complexes.
- The synthesized complexes exhibit good catalytic activity and antimicrobial activity

CONCLUSION

In conclusion, we have described the synthesis and characterization of a series of mononuclear ruthenium(III) complexes containing bidentate ligands and triphenylphosphine/triphenylarsine. The spectral results suggest the presence of an octahedral geometry around ruthenium metal. All the complexes display rhombic EPR spectra of Ru(III) complexes containing distorted octahedral geometry around ruthenium ion. The ruthenium(III) complexes were generated as an efficient catalyst for oxidation

of alcohols. Further, all the new complexes were screened for Ru(III) complexes displays antimicrobial activities.

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