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DRUG DISCOVERY

Spectral investigation of 3,4,5 Tri Methoxy Benzaldehyde-Atomoxetine Cu(ii) & Ru(ii) Schiff base metal complexes & its biological activity

Anjaneya Vasavi GSS[1], Sreeramulu J[2]✉, Subba Reddy GV[3]

A series of New Schiff base Metal Complexes of Cu(II) & Ru(II) were synthesized by the condensation of Atomoxetine with 3,4,5 Tri Methoxy Benzaldehyde. These metal complexes were characterized by Elemental Analysis, UV-VISIBLE, IR, SEM, TG-DTA, Power-XRD & Molar conductance. Molar conductivity data suggested the complexes were non electrolytic in nature. UV-VISIBLE data suggested octahedral geometry of the complexes. The biological activity was identified by E.coli & B.subtils.

INTRODUCTION

Schiff Bases are the condensed products of Amine & Carbonyls like Aldehydes & ketones. These were coined by Hugo Schiff in 1864 [1]. These ligands plays an important role in human welfare and essential in Co-ordination chemistry, the ligands its metal complexes have been used as drugs as tuberculostical [2,3], antibacterial [4, 5], antifungal [6–8], antiviral [9, 10], anti-inflammatory [11], antitumor [12], herbicidal [13] anti cancer, anti diabetic activities [14]and also have important in technical fields like automobile [15] , Photography [16]. These metal complexes also used as conducting and optical organic materials [17].

This paper has provide the information regarding the synthesis and characterization of 3,4,5 Tri Methoxy Benzaldehyde-Atomoxetine Schiff Base and its Cu(II) & Ru(II) metal complexes. These were prepared by conventional method in solvent medium & characterized by Elemental Analysis, UV-VISIBLE, IR, SEM, TG-DTA, Power-XRD & Molar conductance. The power XRD suggested poor crystallinity of the complex [18]. SEM explained the morphology of the ligand & complex [19]. The above data suggested that complexes shown Octahedral Geometry. Biological activity was performed against Escherichia coli & Bacillus sub tills.

METERIALS AND METHODS

All the chemicals purchased were anala A grade. All are used without purification. The used chemicals in these reactions are Atomoxetine, 3,4,5 Tri Methoxy Banzaldehyde, Cu(II) & Ru(II) metals, Con Hcl as a catalyst & Methanol as solvent.

Instrumentation

IR Spectrum of the ligand its metal complexes were recorded on perkin Elmer Spectrometer at JNTUA College of Engineering & Technology Pulivendula. UV-SIBLE Spectrum of the ligand and metal complexes were performed in Santhi Ram College of pharmacy, Nandyal. SEM at Sri Venkateswara University, Tirupathi. XRD at JNT-Univeristy, Anantapur. Molar conductivity at RGM College of Engineering & Technology, Nandyal. Biological activity at Sri Krishna Devaraya University, Anantapur.

Preparation of Ligands Its Metal Complexes

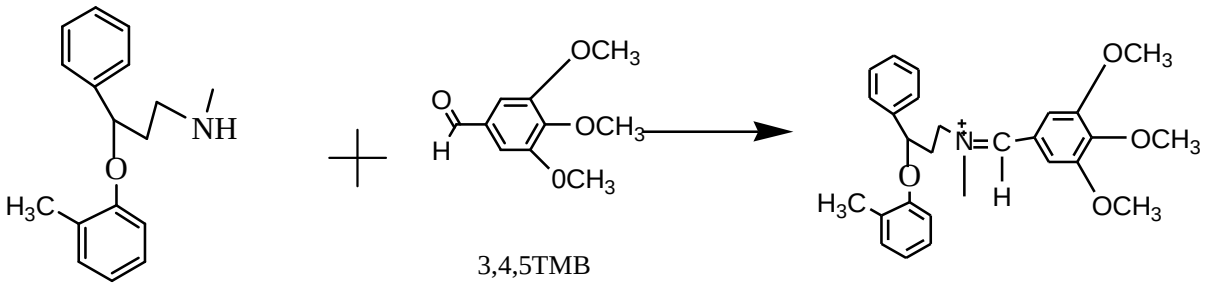
Synthesis of Ligand (Schiff Base)

The Ligand was Prepared by mixing equi molar con of methanolic solution (10ml) of Atomoxetine & 3,4,5 Tri Methoxy Benzaldehyde (10ml) with occasional stirring. This mixture was relaxed for two hours by adding few drops of Con.Hcl, Yellow color solution was obtained when the reaction was completed. This solution on cooling Yellow color sharp needles like crystals were obtained. These crystals were washed & recrystallised with methanol. The percentage yield of the ligand was found to be 68.

Synthesis of Atomoxetine-Copper & Ruthenium metal complexes

The complexes were prepared by mixing an aqueous solution of individual metal ions with the methanolic solution of ligand in round bottom flask separately. These were refluxed for six hours by adding few drops of Con Hcl, leaf Green & brown color solutions were obtained. These solutions were cooled to room temperature; on cooling blackish Brown color sharp needles like crystals& Greenish black color precipitate were obtained. The formed crystals and precipitate were washed with ether and recrystallized with methanol. The percentage yield of the complex was found to be 73 & 78. Colors, Molecular weight, elemental analysis of the complexes were represented in the table I.

[1]Reseaech scholar JNT University, Anantapuramu, AP, India; [2]Professor of Chemistry, S.K University, Anantapuramu, AP, India; [3]Professor of Chemistry, JNTUA College of Engineering & Technology, Pulivendula, AP, India;
✉*Corresponding author:*
Dr. J. Sreeramulu, Mail Id: profjssku@yahoo.co.in



3,4,5TMB

3,4,5 TMB-AT

Table 1 color, MW, %yield & Elemental Analysis

S.no	Name of the Complex	Color	M.W	% yield	% of "C"		% of "H"		% of "N"		% of "O"		% of "M"	
1	3,4,5TMB-AT	Yellow	405.23	68	77.01	**76.99**	7.71	**7.96**	3.45	**3.43**	11.82	**11.80**	-	**-**
2	3,4,5 TMB-AT-Cu	Blackish brown	1109.95	73	69.19	**68.99**	8.17	**8.01**	2.52	**2.38**	14.40	**14.69**	5.72	**5.57**
3	3,4,5TMB-AT-Ru	Blackish brown	2258.15	78	66.93	**66.76**	7.90	**7.78**	2.44	**2.38**	13.93	**13.91**	8.80	**8.69**

Bold values are calculated values

Table II Important IR bands of the 3,4,5 TMB-AT ligand & Cu(II), Ru(II) complexes

Name of the compound	Water - OH	Phenolic-OH	C=N	M-O	M-N
3,4,5TMBAT	-	3363	1626	-	-
3,4,5TMBAT-Cu	3368	-	1607	725	440
3,4,5TMBAT-Ru	3210	-	1621	749	464

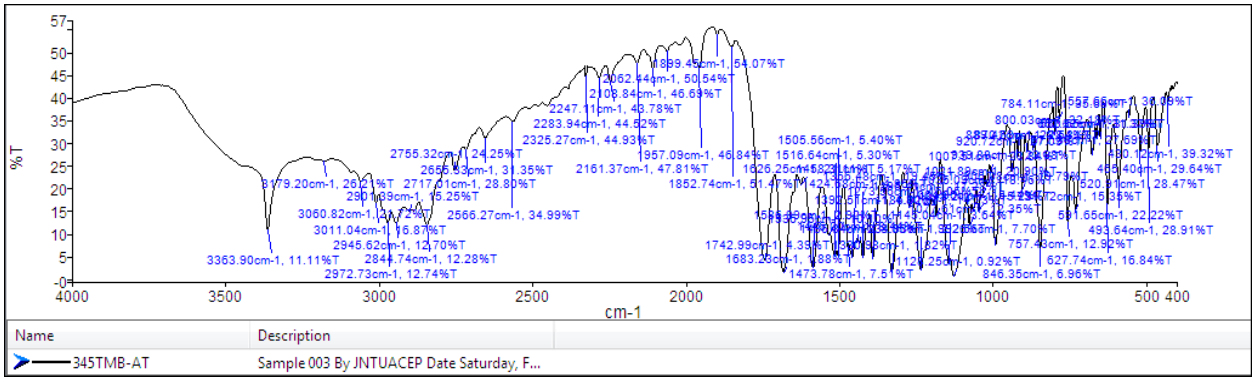


Figure I IR Spectra of 3,4,5TMB-AT Ligand

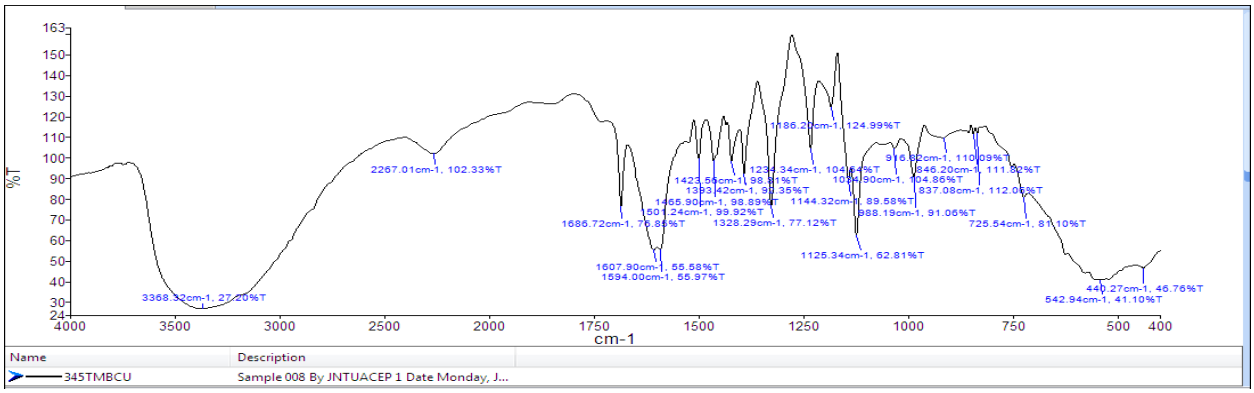


Figure II IR Spectra of 3,4,5 TMB-AT & Ru(II) Metal complex

Table III UV-Spectral data

S. No	Name of the compound	Absorbance
1	3,4,5 TMB-AT	283
2	3,4,5 TMB-AT-Cu	300
3	3,4,5 TMB-AT-Ru	250

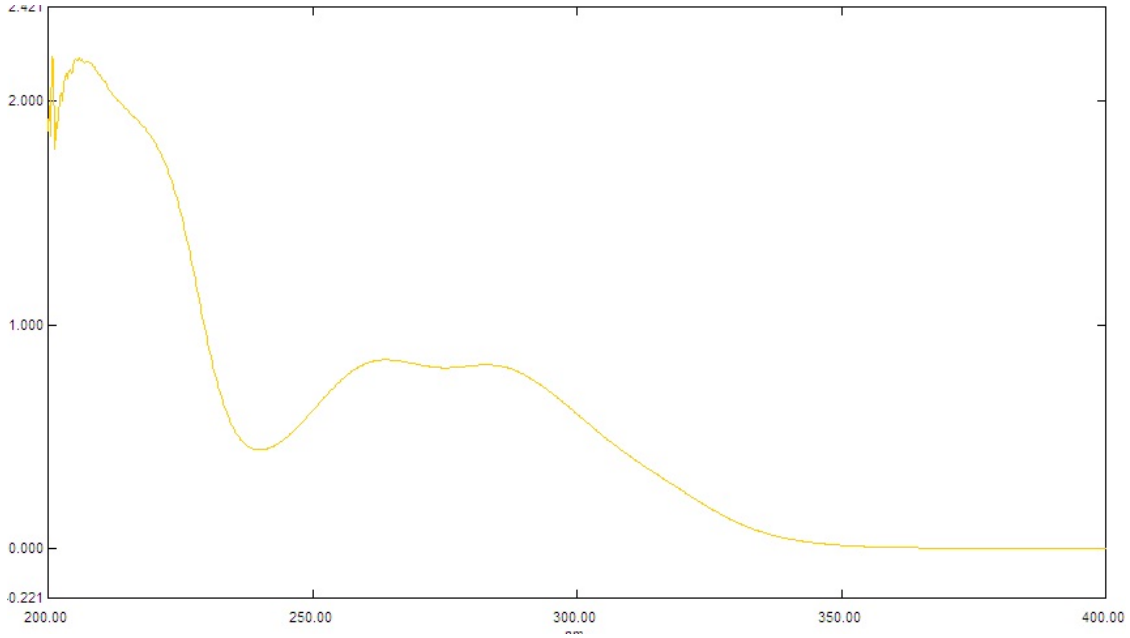


Figure III UV spectral data of 3,4,5 TMB-AT

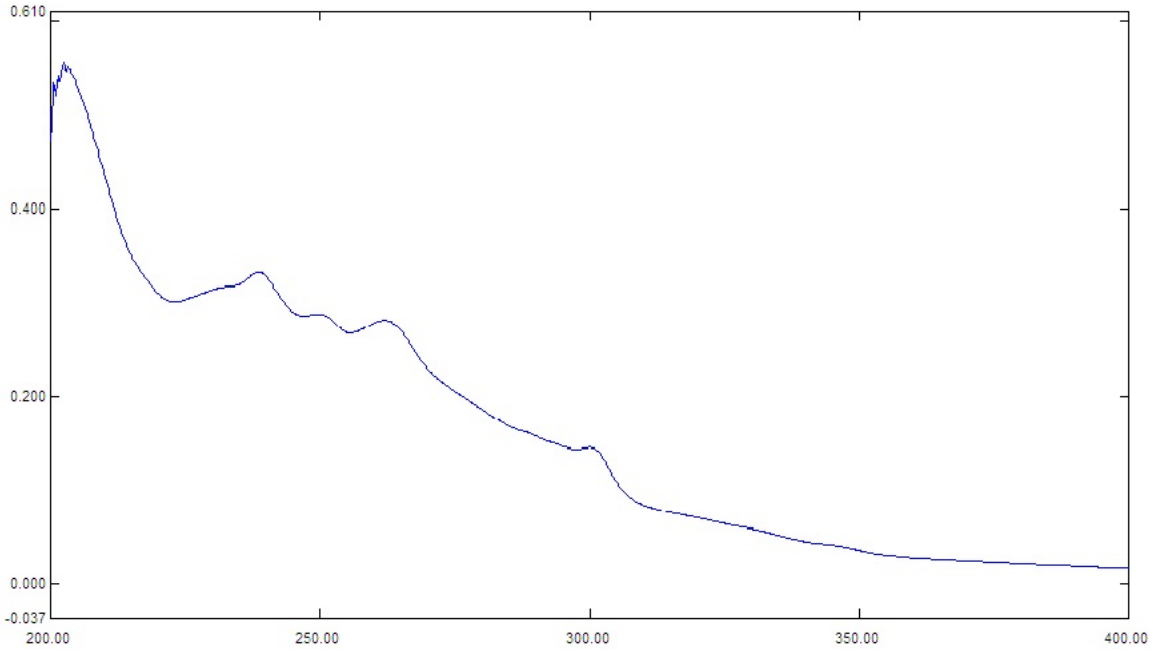


Figure IV UV spectral data of 3,4,5 TMB-AT-Cu

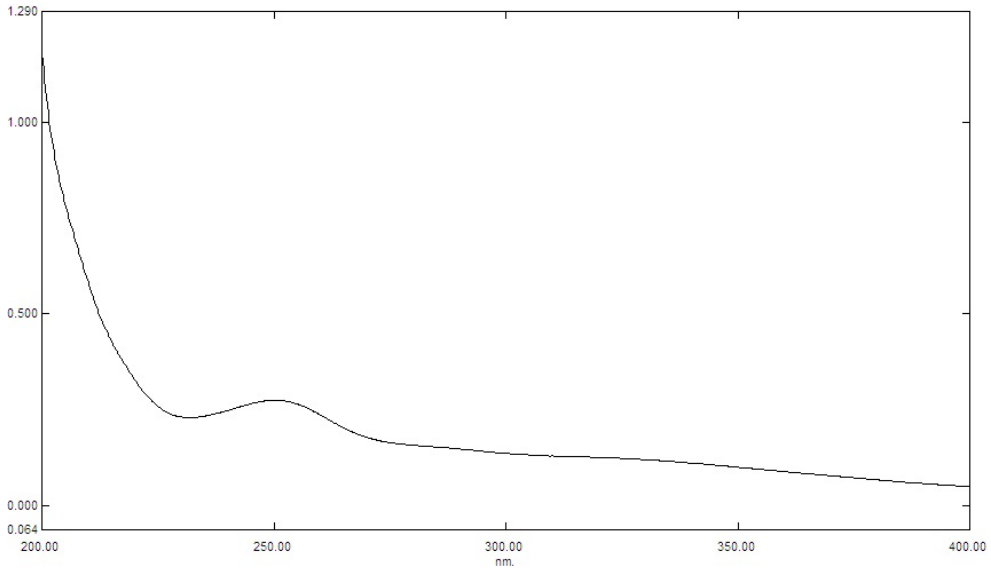


Figure V UV spectral data of 3,4,5 TMB-AT-Ru

TABLE IV X-ray Diffraction study of 3,4,5 TMB- AT-Cu

S.No	d exp	d cal	2Θ exp	2Θ cal	h k l
1	0.0402	0.0394	5.9913	5.9909	1 1 1
2	0.0530	0.0521	7.8972	7.8766	3 2 2
3	0.0701	0.0695	10.4482	10.4475	4 3 1
4	0.0798	0.0790	11.9068	11.9060	5 3 1
5	0.0826	0.0819	12.3833	12.3826	5 2 2
6	0.0980	0.0976	14.9745	14.9739	6 3 2
7	0.1093	0.1086	16.3244	16.3239	7 3 1
8	0.1304	0.1298	19.4978	19.4970	8 4 2
9	0.1347	0.1339	20.1439	20.1931	9 3 1
10	0.1449	0.1440	21.9911	21.9905	10 2 2
11	0.1490	0.1485	22.3203	23.3195	10 2 2
12	0.1512	0.1505	22.6421	22.6416	10 3 2
13	0.1522	0.1515	24.0084	24.0076	10 4 3
14	0.1602	0.1597	25.4420	25.4416	12 2 1
15	0.1696	0.1690	27.3175	27.3168	13 4 2
16	0.1943	0.1938	29.2229	29.2221	14 2 2
17	0.2023	0.2016	30.4653	30.4649	14 4 2
18	0.2082	0.2075	31.3702	31.3694	15 3 1
19	0.2165	0.2158	32.6614	32.6608	16 4 1
20	0.2323	0.2317	35.1176	35.1169	17 3 1
21	0.2534	0.2528	38.4172	38.4166	17 4 2
22	0.2608	0.2599	39.6000	39.5994	18 6 2
23	0.2705	0.2695	41.1240	41.1235	19 6 2
24	0.2828	0.2819	43.0852	43.0846	19 6 2
25	0.3210	0.3204	49.2640	49.2632	19 11 1
26	0.3458	0.3481	53.3650	53.3641	19 17 6
27	0.3714	0.3709	57.6620	57.6615	19 19 3
28	0.4307	0.4299	68.0072	68.0066	19 19 15
29	0.4355	0.4349	68.8686	68.8679	19 19 16

Table V X-ray Diffraction study of 3,4,5 TMB- AT-Ru

S. No	d exp	d cal	2Θ exp	2Θ cal	h k l
1	0.03609	0.03601	5.3720	5.3713	1 1 1
2	0.03944	0.03936	5.8710	5.8701	2 1 1
3	0.04988	0.04981	7.4262	7.4255	3 2 1
4	0.0551	0.0545	8.2183	8.2176	4 2 2
5	0.0635	0.0629	9.4661	5.4654	5 3 1
6	0.0832	0.0826	12.4183	12.4176	6 2 2
7	0.0890	0.081	13.2822	13.2816	8 2 1
8	0.1176	0.1170	17.5773	17.5767	8 3 2
9	0.1237	0.1229	18.4912	18.4902	8 3 3

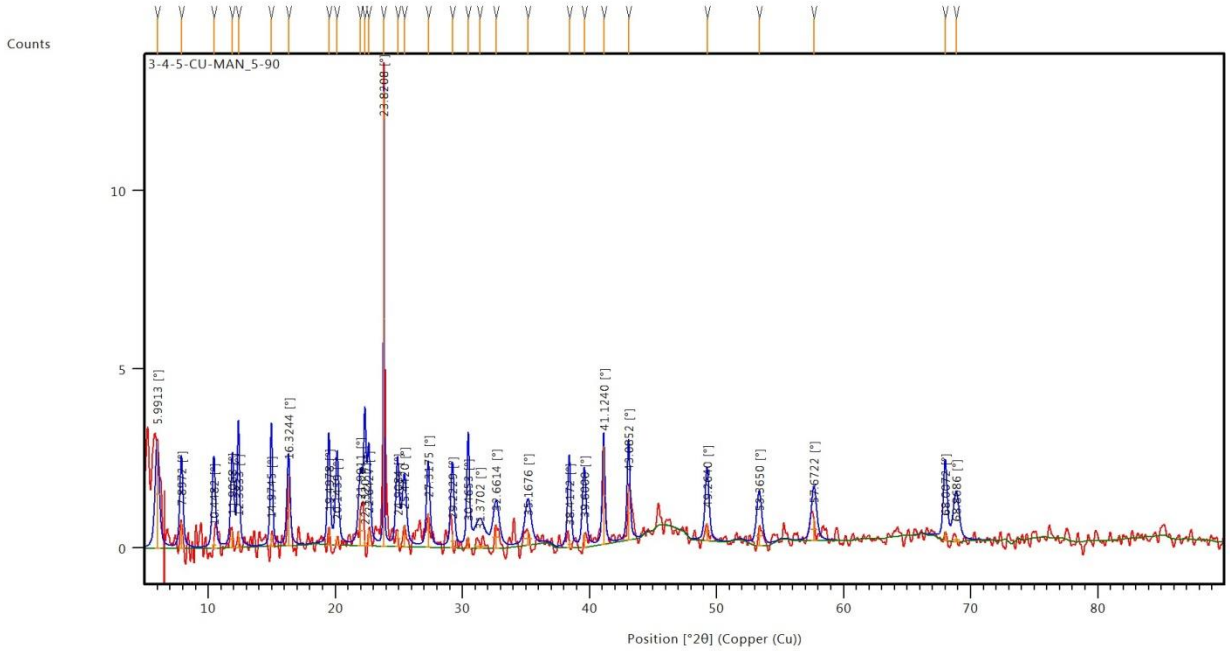


Figure VI XRD spectrum of 3,4,5 TMB-AT-Cu

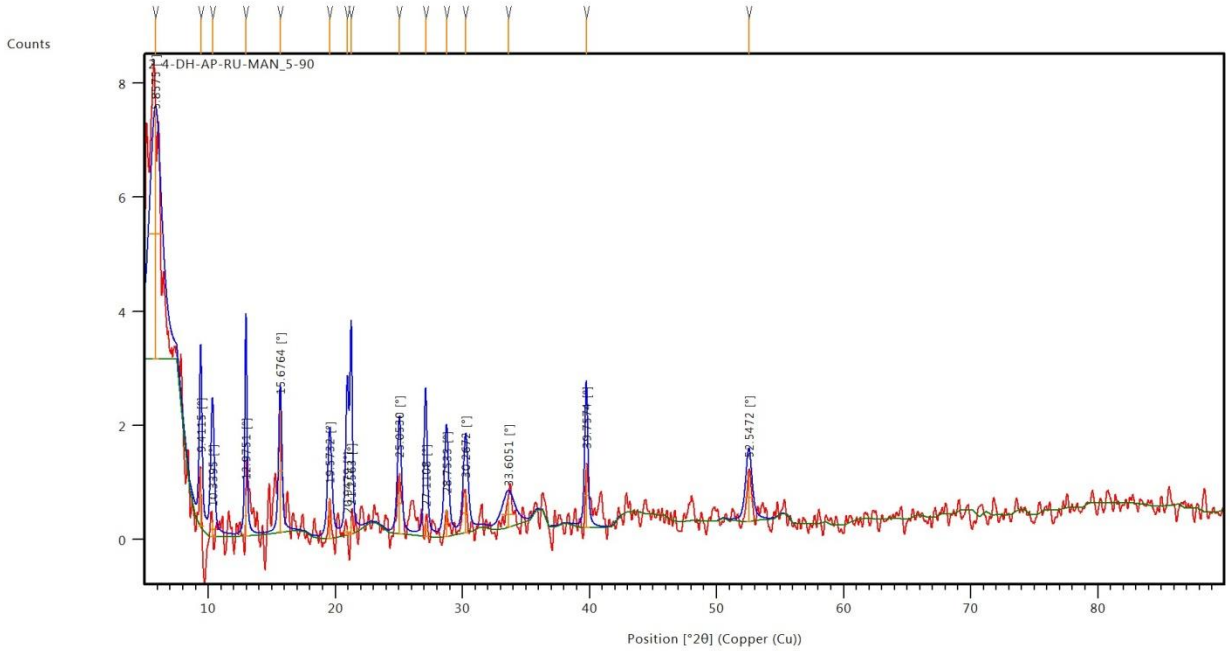


Figure VII XRD spectrum of 3,4,5 TMB-AT-Ru

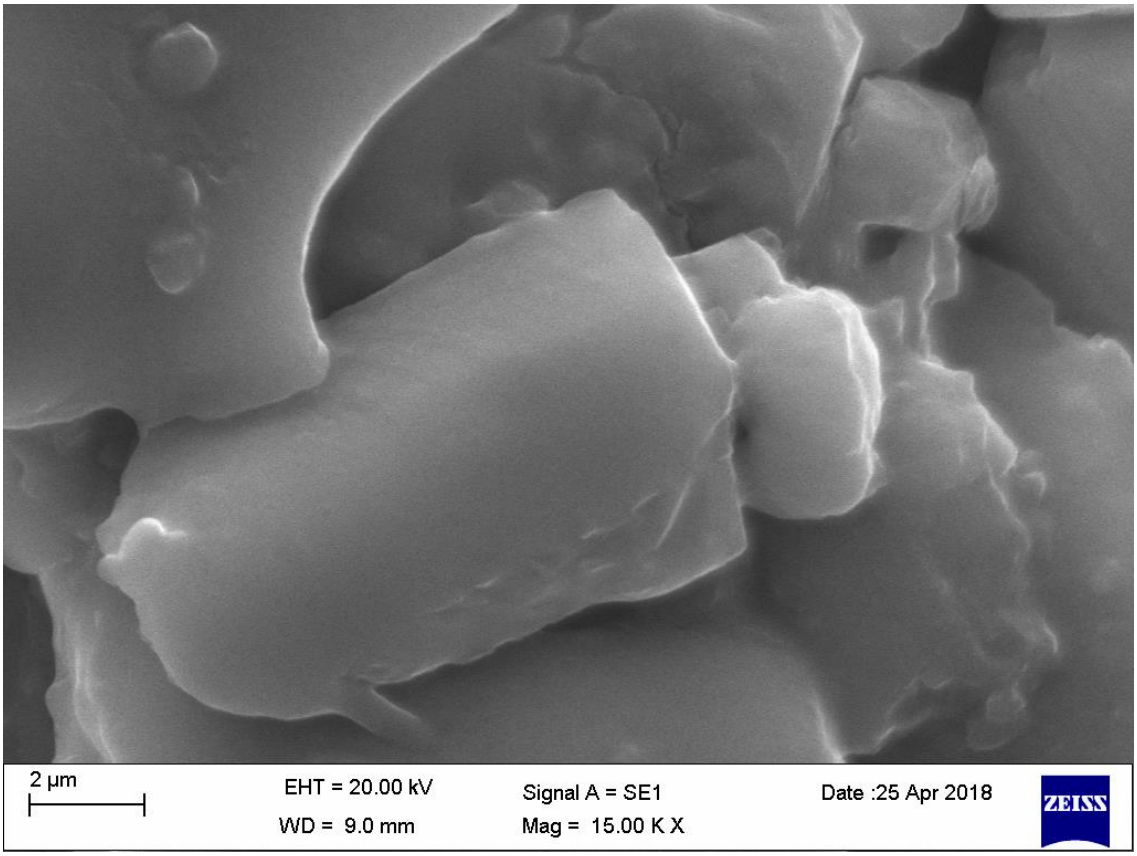


Figure VIII SEM analysis of 3,4,5 TMB-AT-Ru

Table VI Biological activity of the ligand and complexes

S. No	Name of the Compound	E.coli	B.subtils	Klebsiella
1	OVAT Ligand	9.5	10.5	10
2	OVAT-Cu	11	13	12
3	OVAT-Ru	12	14	13

RESULTS & DISCUSSION

IR spectral data

The IR Spectra o f the ligand & its metal complexes were performed in JNTUA college of Engineering & Technology, Pulivendula. IR Spectral data suggested ligand coordinated to metal ion through nitrogen of the imine with electron deficiency of the metal ion and Oxygen of the Banzaldehyde with Metal ion through Co-ordinate co-valent bond [20]. To know the nature of bonding, spectra of the ligand was compared with complexes. A strong band appeared at 1626cm^{-1} shows the formation of Imine group (>C=N). On complexation these were shifted to 1607cm^{-1} & 1621cm^{-1} for Cu (II) & Ru(II) Complexes due to decrease in electron density on Nitrogen atom and the covalent bond formation between Metal Ligand. . The sharp band appeared at 3363cm^{-1} show the presence of Phenolic –OH, this band disappeared in the complexes indicated the deprotonation. The IR data of the ligand & complexes represented in table II, & graphs in Fig I & II.

UV spectral data

UV-Spectral data gives the information about unsaturation and co-ordination between ligands and metal complexes. UV spectral data obtained from Santhiram college of Pharmacy, Nandyal performed using Schimazdu UV-1800 spectrophotometer. The data of the ligand at 230cm^{-1} shows the formation of ligand, this on complexing with Cu(II) & Ru (II) metals the absorbance values at 300cm^{-1} and 250cm^{-1} for Cu & Ru respectively shows the co-ordination of the ligand with metals and this data also suggest octa hedral geometry for both complexes. The absorbance values & graphs represented in the table III & Fig III, IV & V.

Power XRD studies

The power XRD data was obtained from JNTU-Anantapuram. XRD studies have been performed to determine the percentage crystallinity of the complex. Radiation was filled by the metal foil. The diffractions (2Θ) & the calculated miller indices (h k l) values are represented in Tables IV, V & Graph was represented in Fig III. Calculated “D” and difractograms shows a good agreement between metal & Ligand. The difractrograms (2Θ) between 5-70 shows that poor crystalline nature of the complexes [21-22]. XRD patterns are qualitative techniques used to explain the percentage degree of crystallinity.

SEM analysis

Scanning electron micrography (SEM) have been utilized to examine the surface morphology of the complex. The SEM image is represented in Figure VIII. The appearance of the presence of ice cubes, diameter is 2µm at high and low resolutions [23-24].

Conductometry

Conductometry was performed by using digital conductivity meter by using methanol as solvent, the values in between 58-62 $ohm^{-1}cm^{-1}mole^{-1}$ shows non electrolytic in nature. By observing Figure VIII, the spectral information the complexes show octahedral geometry.

Biological Activity

Biological activity of the ligand and their Copper & Ruthenium metal complexes were performed against the bacteria like E.coli, B.subtils & klebsiella shows that complexes shows more bacterial activity than ligands. The reason for increasing activity of the ligands is that the increasing chelation or delocalization of charge over the entire complex compounds therefore increasing liphophilicity of the complex compounds easy pass through the cell membrane and inhibit the growth of micro organisms. Biological activity of the ligand and complexes were represented in the table VI.

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