

Synthesis and Antibacterial Activity of M (II) N-[4 dimethyleamino]benzylidene benzene 1,2, diamine Complex

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Abstract

The M (II) N-[4 dimethyleamino]benzylidene benzene 1,2, diamine complexes (Schiff base) prepared from p- Dimethylaminobenzaldehyde and o-phenylenediamine Metal selected for the preparation of complexes was Co(II), Ni(II) & Cu(II) and three metal complexes were synthesized. The chemical structure of the synthesized metal ligand complexes were confirmed IR, TGA. Elemental and Electronic spectral analysis. The free ligand and its complex have been tested for their antibacterial activity against several bacteria Escherichia coli ,Bacillus megaterium and Pseudomonas aeruginosa,. The antibacterial activity is dependent on the molecular structure of the compound, the solvent used and the bacterial strain under consideration. The antibacterial activity was determined by the Agar Ditch technique using DMF (polar), Acetone (polar) and 1, 4 dioxane (non polar) as solvent. The Ligand and metal complexes showed differential effect on the bacterial strain. The antibacterial activity is dependent on the molecular structure of the compound, the solvent used and the bacterial strain under consideration. In the non polar solvent 1, 4-dioxane the best antibacterial activity was shown by the Ni complex while in polar solvent DMF and Acetone, Cu complex of Schiff base showed best antibacterial activity.

Keywords: Antibacterial activity , DMF, Acetone , 1,4 dioxane P-Dimethyl amino benzaldehyde and o-phenylenediamine

Introduction

Co-ordination compounds exhibit different characteristic properties which depend on the metal ion to which they are bound, the nature of the metal as well as the type of ligand, *etc.* These metal complexes have found extensive applications in various fields of human interest. The nature of a coordination compound depends on the metal ion and the donor atoms, as well as on the structure of the ligand and the metal–ligand interaction [1]. A group of biochemists found their utility and importance in biological systems [2,3,4]. Nitro and halo derivatives of Schiff bases are reported to have antimicrobial and antitumor activities.[5] The newly prepared Schiff base and their metal complexes have been characterized by various spectral techniques. These metal complexes may have enhanced biological activity which may find their importance in the applied medicinal chemistry. . Many Schiff bases are known to be medicinally important and are used to design medicinal compounds.[6,7,8] The complex of Cu (II), Ni (II), and Co (II) with N-[4 dimethyleamino] benzylidene benzene 1,2, diamine have been synthesized.Their antibacterial activity towards some clinically important bacteria was evaluated[9,10,11]

Experimental

Preparation of N-[4 dimethyleamino]benzylidene benzene 1,2, diamine

p- Dimethylaminobenzaldehyde (1.4919 gm 0.1 mol) solution in ethanol and o-phenylenediamine (1.0814 gm 0.1 mol) solution in hot water were taken in round bottomed

flask, 50 ml absolute ethanol was added and the mixture was refluxed for 3 hour. The refluxed mixture was put in ice bath, then orange coloured precipitate was obtained. It was suctioned filtered and washed with distilled water. Schiff base obtained was dried and kept in vacuum dessicator. The pure Schiff base was recrystallized from absolute ethanol

Preparation of Metal Complexes

The complexes were prepared by mixing Schiff base (0.1mol) in hot ethanol solution to (0.1mol) metal chloride salt solution prepared in distilled water. The schiff base solution was added slowly with continuous stirring to metal solution. It was refluxed for 2 hours and after refluxation, the mixture was heated for 10 minutes till the contents was reduced to half. Then the metal complexes precipitated out after being cooled. The precipitate was filtered and washed with the distilled water. All complexes were dried and kept in vacuum dessicator

Antibacterial activity

Antibacterial activity was determined by the Agar-ditch method.[15] The investigated microorganisms were *E. Coli*, *Pseudomonas aeruginosa* and *B. megaterium*. The compounds were dissolved in one of the three solvents (1,4-dioxane, Acetone or DMF) to obtain a final concentration 1mg/0.1 ml. A loop full of the given test strain was inoculated in 25 ml of N-broth (nutrient broth) and incubated for 24 h in an incubator at 37 °C in order to activate the bacterial strain. 28–30 ml of the nutrient agar media was added into a 100 mm diameter Petri-plate. Inoculation was done by the Pour-plate technique. 0.2 ml of the activated strain was inoculated into the media when it reached a temperature of 40–45 °C. The complete procedure of the plate preparation was done in a laminar airflow to maintain strict sterile and aseptic condition. The medium was allowed to solidify. After solidification of the media, a well was made in the plates with the help of a cup-borer (0.85 cm), which was then filled with one of the test sample solutions. Controls were run (for each bacterial strain and each solvent), where pure solvent was inoculated into the well. The plates were incubated for 24 h at 37 °C. The inhibition zone formed by these compounds metal complexes 735 against the particular test bacterial strain determined the antibacterial activities of the synthetic compounds. The mean value obtained for three individual replicates was used to calculate the zone of growth inhibition of each sample.

Result And Discussion

Metal ion plays a vital role in a number of different biological processes through co-enzymatic system. The interaction of these ion with biologically active ligand, for instance in drugs, is subject of great interest. Some biologically active compounds act via chelation, but for most of them little is known about how metal co-ordination influences their activity. The three metal complexes and their respective controls produced different inhibition zones against the tested bacterial strain *Pseudomonas aeruginosa*, *E-coli*, *B-megaterium*. The antibacterial activity of metal ligand complexes in DMF (polar) Acetone (polar) and 1, 4 dioxane (non polar) against *P. aeruginosa* is shown in Fig 1. All complexes showed grater activity in the polar solvent DMF, Acetone compare then the non polar solvent 1, 4 dioxane solvent. In DMF, Acetone the Cu complexes of Schiffbase showed the best activity against *P.aeruginosa*, followed by the Ni (II) complex while in 1, 4 dioxane the best antibacterial activity was showed by the Ni (II) complex, in this study the growth of the gram negative bacterium was inhibited by the Cu and Ni complex. The antibacterial activity against the gram negative bacterium *E-coli* is shown in Fig 2. The antibacterial activity of metal complexes was grater when DMF was used as a solvent. The best antibacterial activity was shown by the Cu complex in DMF and Acetone, while in 1, 4 dioxane only Ni complex showed slight antibacterial activity The effect of the metal complexes on the gram positive bacteria *B-mega* is shown in Fig 3, a different effect of the metal complexes was investigated against this bacterium strain. In DMF, and Acetone the Cu complex showed the best

antibacterial activity was shown followed by Cu, but in 1, 4 dioxane, the maximum antibacterial activity was shown by Ni complex followed by the Co complex, but the minimum antibacterial activity was shown by Co complex of Schiff base.

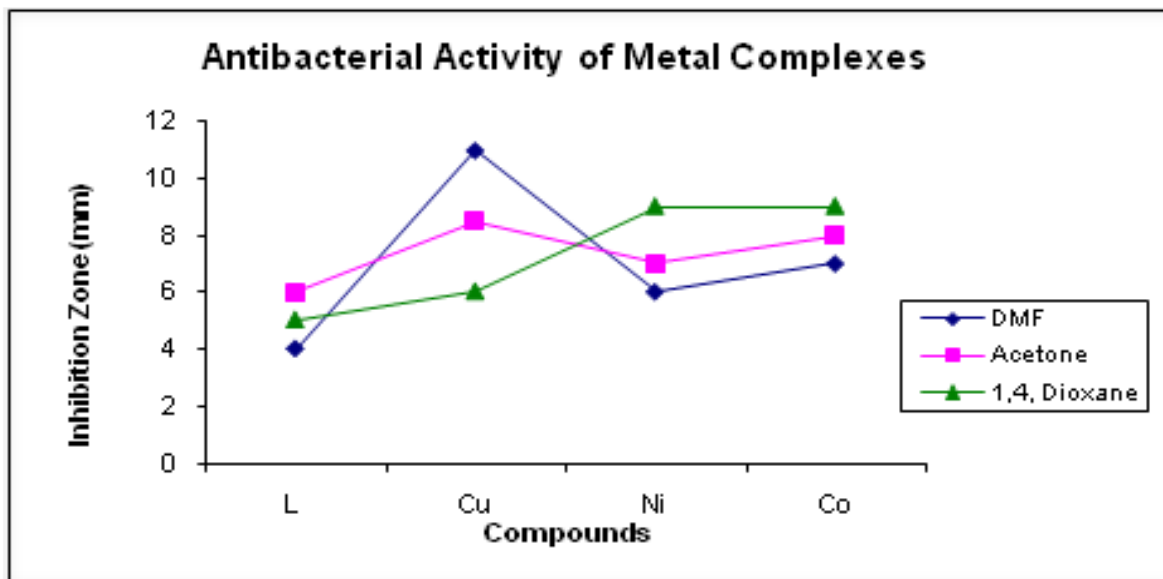


Fig 1. Antibacterial activity of Metal ligand complexes in DMF, Acetone and 1,4-dioxane against *P. aeruginosa*

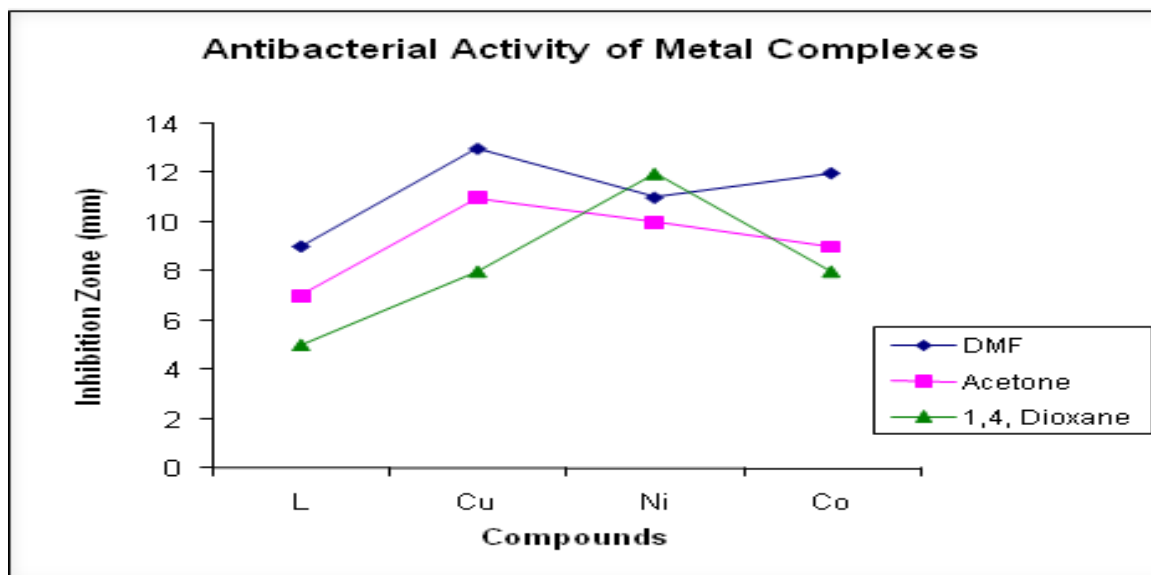


Fig 2. Antibacterial activity of Metal ligand complexes in DMF Acetone and 1,4-dioxane against *E. coli*

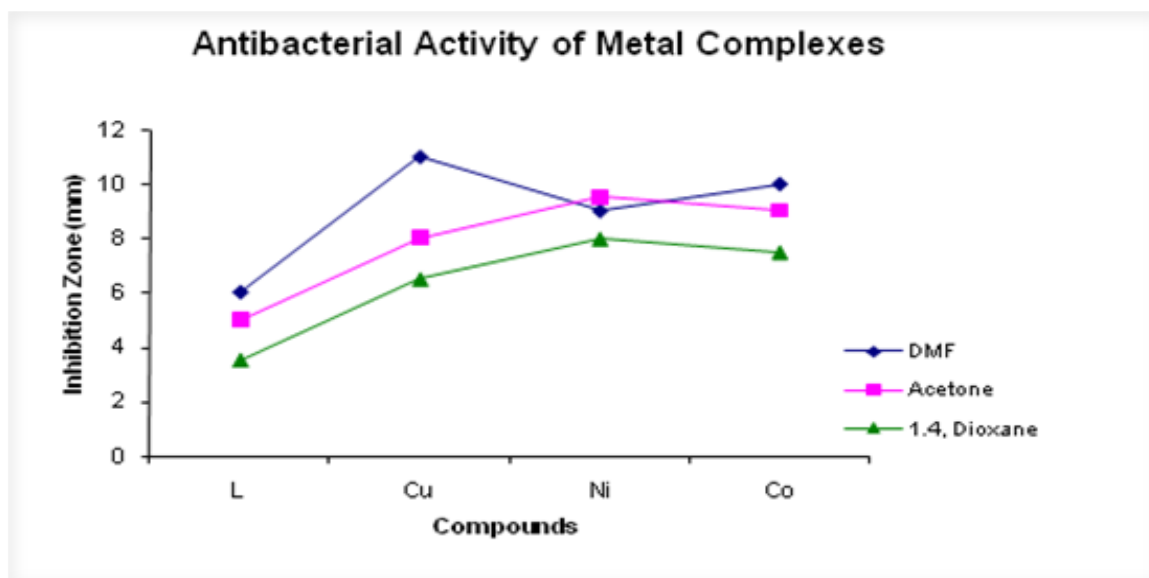
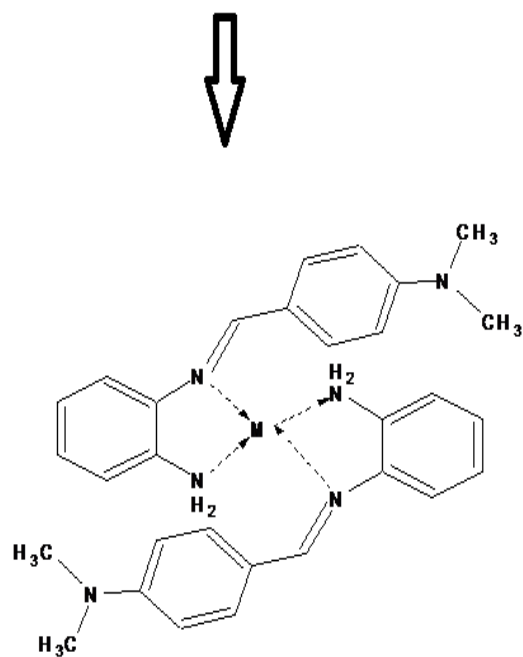
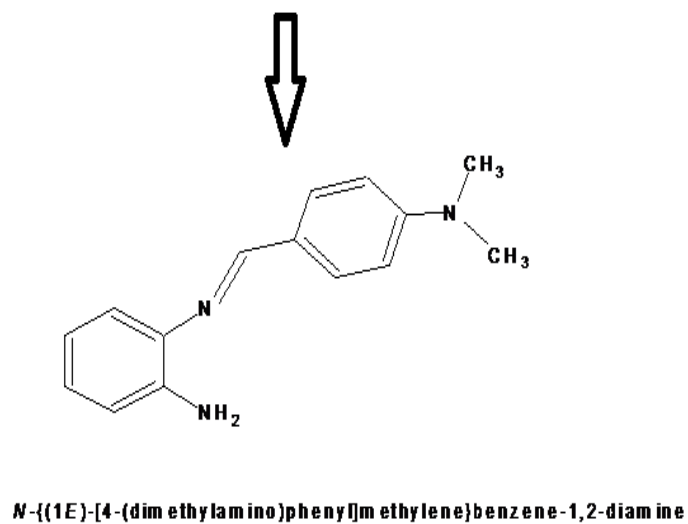
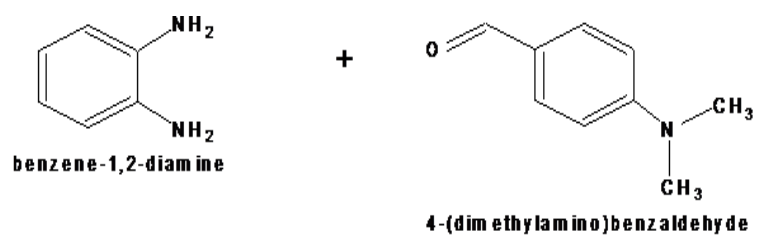


Fig 3. Antibacterial activity of Metal ligand complexes in DMF Acetone and 1,4-dioxane against B -megaterium

This is confirming that antibacterial activity is dependent on the molecular structure of the complex. Reaction of N-[4 dimethyleamino] benzylidene benzene 1,2, diamine and their complexes are as under



[Where M= Cu(II) , Ni(II) ,Co(II)]

The molecular structure of the metal complexes was confirmed by IR, TGA, and Elemental analysis. Their data are given below:

Table: - 1 Analytical and Electronic spectra data of the metal complexes

complexes	colour	Elemental Analysis						Electronic spectra analysis			
		Analysis of elements (%) found (calculated)						Absorption region (cm ⁻¹)	Band assignment	Magnetic moment	
M	C	H	N	O	Cl	μ_{eff} (B.M.)	Geometry				
[CoL ₂].6H ₂ O.Cl ₂	Green	8.24	50.4	6.43	11.74	13.41	9.93	5208 cm ⁻¹	⁴ A ₂ à ⁴ T ₂	1.73	Tetrahedral
		(8.11)	(50.38)	(6.39)	(11.68)	(13.37)	(9.85)	11764 cm ⁻¹	⁴ A ₂ à ⁴ T ₁		
								18867 cm ⁻¹	⁴ A ₂ à ⁴ T ₁ (p)		
[NiL ₂].4H ₂ O.Cl ₂	Brown	8.64	53.09	6.18	12.37	9.42	10.46	5154 cm ⁻¹	³ T ₁ à ³ T ₂	2.83	Tetrahedral
		(8.59)	(52.96)	(6.1)	(12.33)	(9.38)	(10.41)	11494 cm ⁻¹	³ T ₁ à ³ A ₂		
								13333 cm ⁻¹	³ T ₁ à ³ T ₁		
[CuL ₂].2H ₂ O.Cl ₂	Brown	9.81	55.64	5.86	12.97	4.93	10.96	11764 cm ⁻¹	T ₁ ---- ⁴ T ₂	1.73	Tetrahedral
		(9.77)	(55.56)	(5.83)	(12.91)	(4.88)	(10.9)	26315 cm ⁻¹	³ T ₂ ---- ⁴ T ₂		
								broad band			

Table:- 2 IR and TGA spectral data of the metal complexes

Complex	IR									TGA												
	ν -N-CH ₃	ν -C=C-	ν -C=NH	ν -C-H	ν -CH ₂	ν -C-N	ν -M-OH ₂	ν M-N	ν M-N=C	50°C		100°C		150°C		200°C		250°C				
	Schiff base	Schiff base	Schiff base	Schiff base	Schiff base	Schiff base	Metal	Metal	Metal	G	%	G	%	G	%	G	%	G	%			
[Co·L ₂] ·6H ₂ O·Cl ₂	1515 (s)	2920 (m)	3411 (b)	2810 (m)	1368 (b)	1248 (s)	1609 (s)	640 (m)	750 (s)	7.14	1	107.2	3	15	142.9	8	20	150	24	193	27	
[Ni·L ₂] ·4H ₂ O Cl ₂	1519 (s)	-	3382 (b)	-	1374 (b)	1217 (s)	1607 (s)	633 (m)	746 (m)	0.67	0.	8	1	61.08	9	101.8	15	122.1	6	18	156.1	23
[Cu·L ₂] ·2H ₂ O·Cl ₂	1519 (s)	3040 (m)	3338 (b)	2941 (m)	1372 (b)	1215 (m)	1676 (s)	511 (b)	746 (s)	0.64	0.	1	32.37	5	51.8	8	77.7	12	116.5	5	18	

CONCLUSION

For the above results, it can be concluded that amongst the three metals used for complexes formation, in non polar solvent 1, 4 dioxane Ni complex of Schiff base showed the best antibacterial activity but in polar solvent DMF, Acetone Cu complex showed best antibacterial activity. Co complex showed negligible antibacterial activity.

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