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Synthesis and *In-Vitro* Antimicrobial Studies of Transition Metal Complexes of *Dpempa*

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Abstract: *In this work, we presented the synthesis of igand [(2,2'-dibromo-1,2-diphenyethane-1,2-dione)-4-methy aniine](DPEMPA)by the reaction of (2,2'-dibromo-1,2-diphenyethane-1,2-dione) with 4-methy aniine under refux in ethano. The compexes of this igand have been prepared using meta acetates/chorides of Co(II), Ni(II), Cr(III), Cu(II) under refux in ethano-DMF. The products were found to be crystaine soid. The igand is characterized by anaytica, FT-IR, proton NMR spectra data whie compexes have been characterized by anaytica, FT-IR, TGA and magnetic susceptibiity measurements. The compounds were screened for antibacteria activity against some cinicay important bacteria, such asE. coi, S. typhy, S. aureus, P. aeruginosaand K. pneumonieby using nutrient agar medium and antifunga activity against C. abicansand A. niger species by using potato dextrose agar medium.*

Keywords: azomethine group, *in vitro,* antibacteria, antifunga

I. INTRODUCTION

Schiff bases are very important structures for synthetic organic chemistry. Their coordination compounds are known to possess the bioogica activities and inhibit many enzymatic reactions in the ce. Owing to their bioogica activities such as antifunga, antibacteria, antitumor, anti-infammatory, anti-HIV, antidiabetic, antimaaria, and antipyretic, there has been an increasing interest towards the studies of the coordination compounds of the Schiff bases during the past few decades [1-4]. The unique abiity of transition meta ions and their compexes to contro the chemistry of environmenta, industria, and bioogica processes has increased the importance of carifying their mechanistic behavior in simpe and compex chemica processes[5-6]. Recent studies indicated that copper-based Schiff base compounds exhibited better antitumor and antibacteria activities than igands. The copper compexes are aso known to possess good antibacteria activity[7]. Keeping in view the above importance of the compounds, we thought it worthwhie to synthesize and characterize the Schiff base and its coordination compounds with Co(II), Ni(II), Cr(III), Cu(II) ions and these compounds have been studied for their antimicrobia activities.

II. EXPERIMENT

2.1 Materials and Methods

Cobat(II), nicke(II), chromium(III), copper(II) acetate/chorides sats used were of Merck and BDH make. Organic sovents such as absoute ethano, methano, petroeum ether, dimethyformamide (DMF) and dimethysufoxide (DMSO) were of AR grade. The antibacteria activities of the compounds were assessed by using nutrient agar medium and antifunga activity by using potato dextrose agar medium.

Synthesis of [(2,2'-dibromo-1,2-diphenyethane-1,2-dione)-4-methy aniine] (DPEMPA)

The igand DPEMPA was synthesized by condensation of (2,2'-dibromo-1,2-diphenyethane-1,2-dione) with 4-methy aniine by taking ethano as a sovent. It was fitered and dried under reduced pressure at ambient temperature. The purity of igand was checked by eementa anaysis and m.p. It was aso characterized by IR and ^{1}H NMR spectra studies. The purity of the synthesized compounds was monitored by TC using siica ge. (Yied = 79.5 %)

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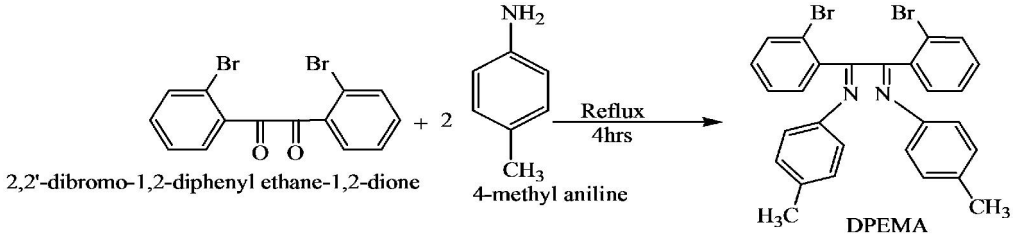
The reaction scheme can be represented as foows,



2,2'-dibromo-1,2-diphenyl ethane-1,2-dione

4-methyl aniline

DPEMA

Synthesis of meta compexes:

A the compexes were synthesized by mixing a ethanoic solution of $M(CH_3CO)_n nH_2O$ and meta haides with the ethanoic solution of Schiff base DPEMPA in a 1:2 moar ratio. The resuting mixture was refuxed on a water bath for 7–9 hrs. A coored product appeared on standing and cooing the solution. The compexes were fitered, washed with petroeum ether and dried under reduced pressure over anhydrous CaC_2 in desiccators. They were further dried in an eectric oven at 60–70°C.

III. RESULTS AND DISCUSSION

The stoichiometry of the igand and its complexes were confirmed by their eementa analysis. The eementa analysis of the igand and its meta complexes show good support with the proposed structures of the igand and its complexes and have been reported in Tabe1.

TABE 1: Anaytical data of DPEMPA and its Complexes

S.N.	Compounds	Sovent used	Coor	Time of Refux (hrs.)	Eementa anayses % found (cacd.)			
					M	C	H	N
1.	DPEMPA	EtOH	Dark red	4.5	--	75.02 (75.27)	6.22 (6.71)	5.78 (5.49)
2.	$[Co(DPEMPA)_2(H_2O)_2]$	EtOH	brown	3	9.87 (9.76)	63.73 (63.68)	6.09 (6.01)	4.70 (4.64)
3.	$[Ni(DPEMPA)_2(H_2O)_2]$	EtOH	Reddish brown	4	9.82 (9.73)	63.74 (63.70)	5.99 (6.01)	4.58 (4.64)
4.	$[Cr(DPEMPA)_2(H_2O)_2]H_2O$	DMF-EtOH	Goden buff	3	8.54 (8.46)	62.48 (62.53)	6.18 (6.23)	4.60 (4.56)
5.	$[Cu(DPEMPA)_2]2H_2O$	DMF-EtOH	Copper eaf	3.5	10.39 (10.45)	63.29 (63.20)	6.02 (5.97)	4.56 (4.61)

Spectra Studies

^{1}H NMR (300MHz, CDC_3, δ in ppm)[8-9]:

12.67 (1H, s, phenoic –OH), 7.64-6.6.37 (7H, m, Aromatic protons), 2.32-2.37 (2H, t, protons attached to Nitrogen), 2.55 (2H, t, benzyic protons), 2.19(3H, s, $-CH_3$).

Fourier Transform-IR (KBr, cm^{-1}):

In a compexes, the band for azomethine group undergoes a shift to ower energy, indicating coordination of azomethine nitrogen with meta ion[10]. This fact is further supported by appearance of some new bands ν(M–N) at 510-578 cm^{-1} and ν(M–O) at 457-490cm^{-1}in the spectra of compexes. In the compex broad band from 3200 to 3600cm^{-1}may be assigned to presence of attice water. In addition to above bands, the IR bands due to pheny ring systems between 1520 and 1566 cm^{-1}which are a most unaffected in the compex have been assigned to aromatic ν(C=C). In a compexes the band for phenoic (C–O) stretching shows a marked shift of 17–25 $cm^{-1}$to higher wave number due to the C–O–M bond

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formation[11] .The band for intramoecuar H bonding is absent in compexes indicating deprotonation of phenoic –OH group and coordination with meta. Bands at 820-850 cm^{-1}may attribute to rocking and wagging modes of the coordinated water. This band is absent in the spectra of Cu indicating absence of coordinated water. It is concuded from the significant shift of free igand ν(C=N) to ower wave number side, increased wave number for phenoic ν(C–O) stretching band in compexes, that bonding of the igand to meta ion is through phenoic oxygen and azomethine nitrogen. The data of IR is tabuated in Tabe 2.

Table 2: Most important IR spectra bands of DHPEPEA igand and its Compexes (cm^{-1})

S.N.	Compounds	*ν*(O-H)/ *ν*(OH--N)	*ν*(C=N)	*ν*(C-O)	*ν*(M-N)	*ν*(M-O)	H_2O
1.	DPEMPA	3237	1625	1290	--	--	--
2.	$[Co(DPEMPA)_2(H_2O)_2]$	--	1603	1387	514	490	3222, 1512, 840
3.	$[Ni(DPEMPA)_2(H_2O)_2]$	--	1591	1360	525	470	3329, 1538, 820
4.	$[Cr(DPEMPA)_2(H_2O)_2]$ H_2O	--	1596	1367	520	457	3300, 1511, 834
5.	$[Cu (DPEMPA)_2]2H_2O$	--	1580	1350	526	485	3439

Antimicrobia Activity

Determination of Minimum Inhibitory Concentration (MIC)

The minimum inhibitory concentration (MIC) is the owest concentration of an antimicrobia compound that inhibits the visibe growth of a microorganism after overnight incubation. MIC of the various compounds against bacteria and yeast strains was tested through a modified agar we diffusion method [12]. In this method, a two-fod seria diution of each compound was prepared by first reconstituting the compound in DMSO foowed by diution in sterie distied water to achieve a decreasing concentration range of 512 to 1 μg/m. 100 μ of each diution was introduced into wes (in tripicate) in the agar pates aready seeded with 100 μ of standardized inocuums (10^6 cfu/m) of the test microbia strain. A test pates were incubated aerobicay at 37°C for 24 h, and the inhibition zones were observed. MIC was recorded for each test organism.

Antibacteria activity

To access the antibacteria activity of obtained compound Agar We Diffusion method [13]was used. This activity was determined by using Muar Hinton Agar [14]. A oop fu cuture of each test organism were inocuated in steriized nutrient agar and incubated overnight to obtain the broth cuture. A the cuture were inocuated on Muar Hinton Agar pate by using sterie cotton swab after swabbing we was punched on media and the different diutions of the compounds were added in to the we with the hep of dropper. After addition of sampe the pate were incubated at 37 °C for 24 hours. After incubation period pates were examined and zone of inhibition were measured.

Antifunga activity

The *in vitro* antifunga assay was performed by the disc diffusion method[15]. The compexes and igand were tested against the fungi *Aspergiusniger*, and *Candida abicans*, cutured on potato dextrose agar as the medium. In a typica procedure, a we was created on the agar medium and nystatin as the contro was inocuated with the fungi. The we was fied with the test soution, which diffuses and the growth of the inocuated fungi is affected. The inhibition zone which deveoped on the pate was measured.

Table 3: Antimicrobia activity of igand DHPEPEA and its compexes (diameter in mm)

Compound	Antibacteria					Antifunga	
	E. coi	*S. typhi*	*S. aureus*	*P. auruginosa*	*K. pneumonie*	*A. Niger*	*C. bicans*
DPEMPA	13	15	12	14	14	18	14
Co- DPEMPA	-	19	14	15	20	19	18
Ni- DPEMPA	-	-	-	08	12	20	19
Cr- DPEMPA	21	10	-	20	21	19	22

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Cu- DPEMPA	13	12	10	12	17	22	20
Amikacin	19	24	23	24	22	--	--
Fuconazoe	-	-	-	-	-	26	24

From the Tabe 3, it is cear that the Co(II) compex shows good antimicrobia activity against *S.typhi*, *K. pneumonia* and *A. niger*. Ni(II) compex shows good antimicrobia activity against *A.niger*. Cr(III) and Cu(II) compexes shows strong activity against *A. niger*and and*C. abicans*

IV. CONCLUSION

On the basis of the anaytica data and other spectra techniques it can be concluded that the compexes of DPEMP. According through its azomethine N and both enoic O atoms. The coordination compounds show significant enhanced antimicrobia activities as compared to the free Schiff base. Therefore, these compounds can be further used in pharmaceutica industry as antimicrobia agents for mankind, after testing its toxicity to human beings.

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REFERENCES

[1]. Konekar H.J.,K. R. Reddy K.R., Isoation And Synthesis Of Bioogicay Active Carbazoe Akaoids. , JHem. Rev. 2002, 102(11), 4303–4428.
[2]. Pandeya S.N., Sriram D., Nath G., European Pharm. Sci., Synthesis, Antibacteria, Antifunga And Anti-Hiv Activities Of Norfoxacin Mannich Bases,2(35), 2000,249-255
[3]. More P.G., Bhavankar R.B., Patter S.C.. Synthesis And Bioogica Activity Of Schiff Bases Of Aminothiazoes. Journa Of Indian Chemica Society. 2001, 78(9), 474–475.
[4]. Yau A.R., Dhande V.V, Bhadange S.G., Aswar A.S., Synthesis, Structura Studies And Bioogica Activity Of Dioxomoybdenum(VI), Dioxotungsten(VI), Thorium(IV) And Dioxouranium(VI) Compexes With 2-Hydroxy-5-Methy And 2-Hydroxy-5-Choroacetophenone Benzoyhydrazone, Russian J. Inorg. Chemistry. 54(4), 2011, 549–554.
[5]. AbduWajid, Mohod R.B., Synthesis and In Vitro Antimicrobia Studies of [1-(2, 4-Dihydroxypheny) Ethanone-B-Pheny Ethy Amine] And Its Compexes, Int. J. Chem. Sci.: 12(2), 2014, 611-617.
[6]. AbeerTaha, AbeerKarim,Physicochemica Studies And Bioogica Activity Of Mixed igand Compexes Invoving Bivaent Transition Metas With A Nove Schiff Base And Gycine As A Representative Amino Acid, European Journa Of Chemistry 5(2), 328-333, 2014.
[7]. Chetana P., VibhaVinayakumarBhat, Hetero-Binucear Compexes Of anthanum (Iii) Using Bridging N,N-Bis(2-Pyridymethy)Oxamide And Termina 1,10-Phenanthroine: Synthesis, Characterization And Bioogica Evauation, Internationa Journa Of Pharmaceutica Sciences And Drug Research, 10(6), 460-73, 2018
[8]. DyerJr, "Appication Of Absorption Spectroscopy Of Organic Compounds", Phi earning Private imited, New Dehi; (2010).
[9]. SiverteinRm And Webster Fx, "Spectroscopic Identification Of Organic Compounds", 6th Ed., John Wiey And Sons, Inc, New York; (2011).
[10]. Agrawa N. K., Aneja K. R., Dhimanin R., Vitro Antimicrobia Activity And Phytochemica Studies Of TerminaiaChebua Against The Microbes Isoated From Fruit Juices,Journa Of Microbioogy, Biotechnoogy And Food Sciences, December, 5(3):243-247, 2015.
[11]. Mishra A.P., Mishra R. K. and Shrivastava S. P., Structura And Antimicrobia Studies Of Coordination Compounds Of Vo (II), Co (II), Ni (II) And Cu (II) With Some Schiff Bases invoving 2-Amino-4 Choropheno, J. Serb. Chem. Soc. 74(5), 523–535, 2009.
[12]. British Pharmacopoeia-Ii, Bioogica Assay and Tests, The Stationary Office td., ondon; A-205, 1998.
[13]. Mueer J.H. And Hinton J., Proc. Soc. Exp. Bio. Med., 48, 330, 1941.
[14]. Prescott S.C., Duan C.G., Industria Microbioogy, 3rd Ed., Mcgraw Hi, Koyakesha, 519, 1949.

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[15]. Abdu Wajid And Mohod R. B., Synthesis Of Amino Acid Based Schiff Base And Its Compexes As Microbia Growth InhibitorsRasayan J. Cem., ,6 (4), 284 – 287, 2013.