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Fungicide Activity of New Complexes for Fungi *Rhizoctonia solani*, *Fusarium solani* and *Macrophomina phaseolina*

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***Abstract*—**This work includes the preparation and diagnosis of nine complexes which prepared interaction copper chlorides ,cobalt, nickel, and cobalt nitrate beside Schiff base salts obtain from N- dimethyl benzaldehyde with numerous amino acids like glycine and L-serine. we found that metal ions consistence to complexes as bidentate manner, condensation occurred via azomethine nitrogen with carboxylic oxygen atoms. Analytical data showed that all cobalt complexes1,2,3 having an octahedral geometries with the formula $[Co(Bx)(H_2O)_2(NO_3)]$, Bx= (NaBg or NaBph), nickel complexes4,5,6,7 having tetrahedral geometries with the following formulas ; $[Ni(Bz)(H_2O)_2]Cl$,where: Bz =(NaBg or NaBph or NaBs or Bt and $[Co(By)(H_2O)_2]Cl$,where By =(NaBs or NaBt), while copper complexes8,9 having square planer geometries $[Cu(Bz)(H_2O)_2]Cl$, Moreover , Schiff base complexes elevated for antifungal activity in Vitro against three types of important pathogenic fungi namely :*Fusarium solani*,*Macrophomina phaseolina and Rhizoctonia solani*. All complexes gave significant inhibition against these pathogens especially4,8,9 complexes . The inhibition percentage of complex 9 with concentrate 100ppm *against R solani,. F.solani* was100%,99,3% and 75ppm had the same antifungal activity against *R .solani* and *F.solani*. 100% -93.9% respectively Moreover the percent inhibition increase to 64.7% *with complex* 8 *at* concentration 75ppm against *M..phaseolina*.

Keywords— antifungal activity, Schiff base complexes

I. INTRODUCTION

Great attention to the chemistry of complexes containing azomethine group (–C=N-) has been paid lately , Those complexes has a wide application in the field of industrial chemistry , pharmaceutical and Agriculture[1,2]. Synthetic flexibility , ,selectivity and sensitivity towards transition and non-transition metal atoms, all those factors made Schiff bases important [3] The interaction between amino acids and aldehydes or ketones produce Schiff bases which have considerable importance in the field of metalloenzymes and other biological activities [4,5]. They are present in keto-enol forms and can form different types of complexes[6-9]In the present work, Preparation and diagnosis of Schiff base complexes synthesis via N- dimethyl benzaldehye and amino acids have been done via physicochemical and spectral technique … Silver dressing for burns therapy , bismuth drugs for ulcers medicating ,zinc exorcized cream and metal complex having a bond between two metal ions for HIV drugs ,All of these use today as chemotherapeutic for many microbial infection [7]. *Aspergillus niger*, *Candida albicans* and *Escherichia coli* were inhibited in vitro by many Schiff base complexes[8,9]. The antifungal activity of the different complexes had been studied in college of science /Department of Biology University of Mosul and performed sing the diffusion agar technique[7,8].Fungi as Candida albicans inhibited by Schiff bases arise via benzaldehydes and 3,3-diaminodipropylamine in concentration 24mg/ml which consider as favorable antifungal factor[10] Phytopathogenic fungi for example: *A. brassicae*, and *A. brassicicola* that affect some crops like rape, radish, broccoli and cauliflower so Schiff bases derived from N-Salicylidene -2-hydroxyaniline inhibited the growth of them by 67-68% respectively at the concentration of 500 ppm [2].

II. RELATED WORK

An essential group of ligands that correlative to metal ions are derivative from an amino and carbonyl complexes (Schiff bases) show effective bind to DNA and DNA grooves, so restrain the growth of Stomach cancer cells[11]. Rresearchers determined the oscillating beams in the water complexes at (650 - 880 cm - 1) For the inorganic salts of the water harmonic[12]

Series of complexes of Schiff's bases with large asymmetric rings to formula[M(L)Cl], M (II) = Co , Ni , Cu were prepared and By the condensation reaction of the substituents 1-amino-5-hydrazino-1,2,4 (N tetragonal ligand)[13] Tyagi,2014[14] isolated and reported three octahedral complexes from copper (II) nickel, (II) cobalt (II) ions(type derived from Aminoantipyrine and dibenzoylmethane with ethylenediamine). The ligand was

prepared through the(II) beta-diketone condensation reaction of compound- 1 (3-Dione, 2-hydroxyphenyl , 1 propane 4-chlorophenyl) and the Amen binary ortho-phenylene [15] Schiff bases also have broad biological activities, including anti-fungal, anti-bacterial and anti-inflammatory action [16,17,18,19,20,21 22] and therefore, the complexes of Schiff bases have a higher biological activity than the ligands that make up these complexes [23] as it has been observed that the conversion of biologically active compounds into lipid bases improves the biological activity of those Compounds, especially when using amino compounds such as amino acids.[24]

III. METHODOLOGY

Chemical Studying

All compounds and dissolving agents utilized of analytical process. The metal salts were get as pure sample from Aldrich, Fluka, or(B.D.H).

Preparation of Schiff base salts have been made just as literature method [10]

Dissolving per amino acids in 20 ml of 1:4 (distal water: ethyl alcohol) then mixing with C9H11NO (0.0 1 mol) added CH3COONa(0.01mol) utilizing water bath at 50 0C for one hour, measuring PH after Cooled mixture and evaporated it for one night to collect precipitate which filtered and washed with (diethyl ether plus ethanol in rate1:1)dried with Calcium chloride anhydrous.

Preparation of the Metal Complexes in Molar Ratio (1:1) Metal : Ligand

0.01mol of Schiff base salt was dissolved in 20 ml ethanol then mixed with 0.01 mol. cobalt (II) chloride or nickel (II) chloride or copper(II) chloride and cobalt (II) nitrate in 10ml of hot ethanolic solution , added(0.02 mol) sodium acetate in form aqueous solution (drop by drop).By stirring(one hour, 50c0) the combination has been retracted on half then cooling, fixed pH and volatized, filtered, washing by C2H5OC2H5 and dried over CaCl2.

Analytical and Physical Measurements:

SMP30 melting point equipment was utilized to estimate melting points (M.P) temperature or disintegration (d) of prepared compounds. Metal contents have been delineates by utilizing atomic absorption Sons AAGB Scientific Equipment, Dissolution complexes via concentrated nitric acid . CHN analyzer elemental vector, model EA 3000 V.3.0 single Euro using to determine elemental analysis. Utilize 10–3M dimethylformamide solution(25°) and BC 3020 professional Bench top conductivity the molar conductivity to complexes had been measured. IR spectra reading of compounds were recorded via Shimadzu FTIR-Tensor 27 -Burker Co. Germany 2003 as KBr pellets in the range 400-4000 cm-1 .Spectrophotometer UV- visible (Shimadzu U.V.210) measured IR Spectra in dimethylformamid solvent for 10-3 M complexes (25° C) with 1cm quarts cell in limits (200-1000) nm. Magnetic sensibility of the complexes were evaluate by Faraday manner apply Bruker –BM6 at 25°C.

Biological studding

Antifungal activity with prepared complexes

Studying of nine complexes to get 100ppm of each fungal genus were assayed by disc diffusion method(7,8,9)by dissolved 0.1g of each complexes in 1.5ml of Dimethyl sulfoxide(DMSO) to 100ml of culture media (PDA) being that the petri dishes were left to solid also prepared 12.5,25 50,75ppm for each active complex.

Inoculation of tested fungi

Each of Petri dishes inoculated with 5mm diameter disc of fungal mycelium After the last step taken from pure culture7days old, Incubation for 6 days, the radially mycelia growth were measured, then calculating inhibition percentage by equation: Inhibition percentage

$$=\frac{\text{average growth in control}-\text{average growth in treated}}{\text{average growth in control}} \times 100$$ (three replicates for every treatment).

IV. RESULTS AND DISCUSSION

In Table 1 the molar conductance values of the complexes 1&2 in10-3M DMF are 22& 20 ohm -1 cm2 mol-1 refer non electrolytic in nature [25]while complexes 3 -9 are in limits 65-92 ohm -1 cm2 mol-1 indicating a 1: 1 electrolytic nature of these complexes[26]

Table 1: Analytical data of the complexes

No.	Complexes	Ω $Ohm^{-1}.cm^2mol^{-}$	% Analysis Calc. (Observ.) M%	C%	H%	N%
1	$[Co(Bg)(H_2O)_2(NO_3)]$	22	16.48 (16.65	36.87 (37.00)	4.74 (4.93)	1173 (12.00)
2	$[Co(Bph)(H_2O)_2(NO_3)]$	20	13.16 (13.00)	48.78 (49.00)	5.13 5.00))	9.39 (9.50)
3	$[CoBt)(H_2O)_2]$ Cl	85	13.51 (13.75)	49.48 (49.67)	4.58 4.81))	9.62 (10.00)
4	[Ni $(Bg)(H_2O)_2]$ Cl	78	17.70 (17.66)	39.81 (39.77)	6.33 (6.41)	2.66 (12.68)
5	$[Ni(Bph)(H_2O)_2]Cl$	92	13.93 (13.87)	51.28 (50.55)	5.46 (5.50)	6.64 (7.00)
6	$[Ni(Bs)(H_2O)_2]$ Cl	74	16.25 (16.65)	39.86 (39.52)	4.42 (4.80)	7.75 7.47))
7	$[Ni(Bt)(H_2O)_2]$ Cl	65	13.45 (13.22)	49.51 (49.20)	4.58 4.25))	(6.41) 6.33))
8	$[Cu(Bg)(H_2O)_2]Cl$	69	18.77 (19.00)	39.34 (39.00)	5.06 5.11))	8.34 (8.37)
9	[Cu $(Bph)(H_2O)_2]$ Cl	90	14.80 (15.00)	50.76 (50.41)	5.40 (5.50)	6.58 (6.63)

Slight deviation to lower frequency to all complexes refer correlative of the Schiff bases over azomethine nitrogen atom [27,28]. Covalence Schiff bases via COO-group was related to this value in monodentate conduction [29,30].

The table 2 show three (N-O) stretching bands. A virtually estimate the covalence of nitrate group rate of ~220 cm-1 for complexes is reported as (v5-v1), Both relative energies and number estimate the coordination of nitrate combine frequencies (v5-v1) in the infrared spectrum display strong covalence for the metal-nitrate bonding and can be utilize for characterizing numerous covalence forms to the nitrato group[31].

In the current complexes separation of 190-180 cm-1,and the nitro groups show as bidenate. Stretching vibration OH of water reason feebly to intermediate broad bands in (3425-3206) cm-1 for aqua complexes [32]. Bending vibration of water due to a sharp shoulder at (1513-1590)cm-1 . OH rocking vibrations lead to coordinate water molecules through manifestation of added effective and acute band at 800-875cm-1 [33]. New bands at (410-510) and (518-589) cm-1 to all complexes were recorded and refer to the stretching form of M-N and M-O sequentially, [34]

Table 2: Infrared spectroscopy bands (cm^{-1}) of the complexes

No.	$\nu_{C=N}$	$\nu sCOO$	$\nu asCOO$	$\nu M\text{-}N$	$\nu M\text{-}O$	ν_{OAH2}
1	1595	1364	1514	451	535	3425, 865
2	1590	1411	1513	433	529	3306, 841
3	1585	1408	1506	440	589	3417, 822
4	1607	1400	1510	510	588	3228, 810
5	1579	1363	1512	433	529	3206, 830
6	1582	1389	1510	480	525	3400, 875
7	1583	1375	1509	491	543	3317, 800
8	1590	1360	1500	472	535	3350, 800
9	1578	1355	1512	410	518	3240, 840

Ultraviolet spectra , d-d spectra and charge transfer spectra of the complexes in DMF solution have been giving the electronic spectra Table 3. An effective magnetic moment of complex 4 equals to 4.35 B.M., which lies in the domain(4.1-4.8) B.M. ,for most of the mononuclear Co (II) complexes in tetrahedral environment at room temperature and theses values reveal that these complexes possess tetrahedral geometry and indicating monomeric nature[35,36,37] The magnetic moment obtained for complexes 1 ,2 are 4.54, 4.61 B.M. respectively due to orbital contribution and these values agree with high spin octahedral configuration[38] . While Ni(II) complexes 4-7 exhibit ν_2 band at 14492-14880 cm^{-1} that can be ascribe to the: $^3T_1$ (F) $\rightarrow$ $^3T_2$ (P), transition in a high spin tetrahedral shape [39]. Magnetic field for copper (II) complexes 8-10 are at 1.96-1.75 B.M., corresponding square planer environment circulate the Cu (II) ion[40].

Table 3: Electronic Absorption Spectra and Geometry of complexes

No.	μ_{eff} (BM)	Electronic Spectra cm^{-1}	Geometry
1	4.54	9607,10929,16241	Oh
2	4.61	9460,12224,15015	Oh
3	4.35	13250	Td
4	6.13	14492	Td
5	5.99	12369	Td
6	1.64	15300	Td
7	1.96	14880	Td
8	1.86	14531	Sq .pl
9	1.75	17809	Sq .pl

Sq.Pl=Sequare planer , Td=tetrahedral , Oh=Octahedra

In biological results as shown in(table 4)the most effective complexes are4,8,9 in concentration 100ppm against *R.solani* ,so the percentage inhibition raised to 100% with complex9 in different concentration against the same species.The% inhibition of *F.solani* were(12.5,99.3%) for 6,9 complexes respectively (figure 1). Table(5) shown that inhibition of *F. solani* decrease with small concentration of 4,8complexes .while complex 9was most efficient with %inhibition 93.9,67.6% in 75,50 ppm and same complex shown 100% inhibition against *R. solani* in both 75,50ppm as in (table5). *M.phaseolina* inhibited by complex 8 in two concentration (figure 2) We found in previous research[41] that 70% of topchem wp(Thiophanate methyl)inhibition increase to 100%in 100mg/l against *R.solani* also popchem(hymexazol) recorded most higher inhibition 100% in150mg/l against *R.solani* . Remarkable efficacy to Schiff base of (E)-N-(4-thiophen-2-ylmethyleneamino phenylsulfonyl) Acetamide metal complexes as antifungal for (*Aspergillus fumigatus* and *Candida albicans*)[42].. Inhibition rate of complex Cu (II) reached to 46% against *Candida Albicans* but does not influence against *Aspergillus Niger* [43]. Metal-imine compounds had antifungal effect against *Helminthosporium gramineum* which responsible of leaf stripe in some economic plants[44].

Table 4: Efficiency of prepared complexes in (100ppm)to inhibition percentage of Rhizoctonia solani, Fusarium solani,Macrophomina phaseolina

No.	% Inhibition *R.solani*	% Inhibition *M.phaseolina*	% Inhibition *F. solani*
1	3.75	1.25	26.1
2	55.87	3.12	8.37
3	52.5	4.25	3.12
4	97.5	61	32.3
5	87.12	9.25	9.37
6	39.62	16.75	12.5
7	0	10.5	9.37
8	99.3	88.7	32.8
9	100	3.12	99.3

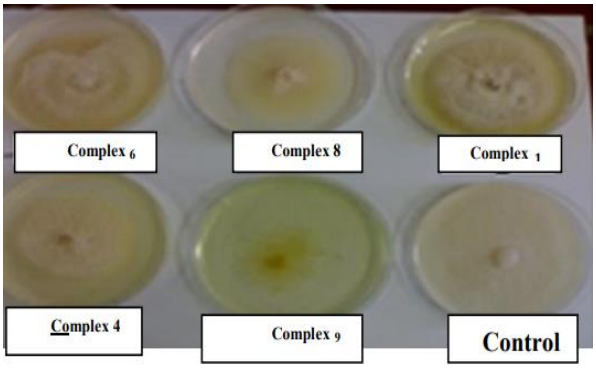


Figure 1. Effect of (1,4,6,8,9) complexes100ppm against *F.solani*

Table5:Effect of 4,8,9 complexes with different concentrations against fungi

Fungi	N.o	% Inhibition			
		12.5	25	50	75
M. phaseolina	4	0	8.7	14.7	46.2
	8	0	8	35	64.7
	9	0	0	0	0
R. solani	4	18.4	29.2	95	96,9
	8	53,2	75.3	98.1	98.7
	9	66.1	93.8	100	100
F.solani	4	0	6.0	12.5	25.31
	8	2.9	7.4	13.29	26.47
	9	49.8	51.2	67.6	93.9

Note:12.5,25,50,75 refer concentration in (ppm)

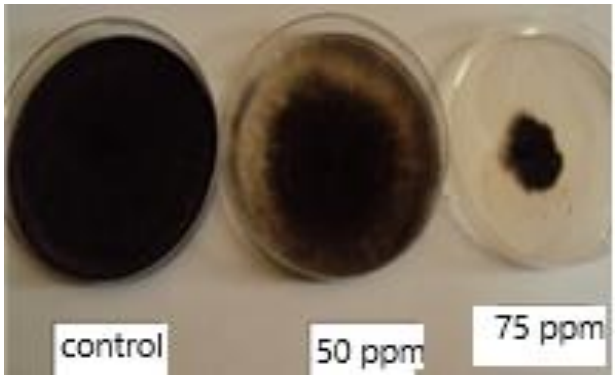
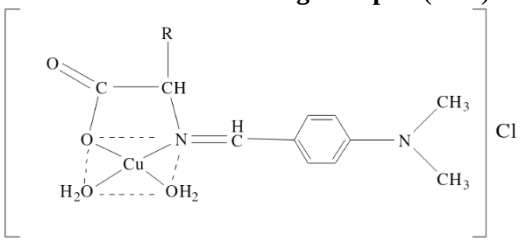


Figure 2. Effect of complex (8) in (50,75ppm) against *M. phaseolina*

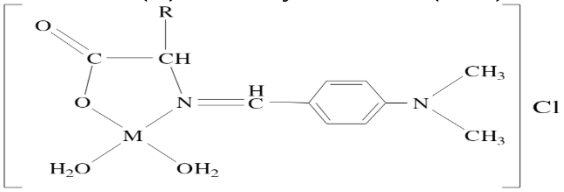
IV.CONCLUSION

All nickel complexes and two cobalt complexes having tetrahedral geometries with the following formulas ; $[Ni(Bz)(H_2O)_2]Cl$,where: Bz =(NaBg or NaBph or NaBt or NaBs and $[Co(By)(H_2O)_2]Cl$,where By =(NaBs or NaBt), while copper complexes having square planer geometries $[Cu(Bz)(H_2O)_2]Cl$. Moreover , cobalt complexes having an octahedral geometries with the formula $[Co(Bx)(H_2O)_2(NO_3)]$.

square planer complexes: $[Cu\ (Bz)(H_2O)_2]\ Cl$
B Z= BS or Bt or Bg or Bph : (9-12)



tetrahedral complexes: $[M\ (BH)(H_2O)_2]\ Cl$
M= Ni(II) ,BH=Bz= Bg or Bph or Bs or Bt
M= Co(II) , BH = By=Bs or Bt , (1,4-8)



All complexes gave significant inhibition against three pathogens *Fusarium solani*,*Macrophomina phaseolina*, *Rhizoctonia solani*. The percent inhibition growth of the fungi shows greater efficacy for the complexes 4,8,9 with four concentration (12.5,25,50,75 ppm). The complex 9 at 75ppm had the best antifungal activity against *Rhizoctonia solani* 100%.

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