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Synthesis, Spectral Characterization and Invitro Antifungal Activity of Novel Metal Complexes

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ABSTRACT

The novel ligand, 2-(6-(2-hydroxy phenyl)-[1,2,4] triazolo [3,4-b][1,3,4] thiadiazol-3-yl)-N-phenyl benzamide (TTS) (2) was prepared from 2-(4-amino-5-mercapto-4H-1,2,4-triazol-3-yl)-N-phenylbenzamide (1) with salicyldehyde. The transition metal ions were used to synthesize various metal chelates of ligand (2).The synthesized ligand and its metal chelates were analyzed by elemental analysis, spectral study, ratio of M:L and magnetic characterization. Antifungal activities of the entire novel synthesized ligand and its metal chelates were screened.

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Introduction

In current time, the number of research work was carried out on metal complexations. [1] Coordination chemistry shows various organic compounds and their metal complexes [2-4] Triazole containing organic compounds demonstrates diverse medicinal with biochemical activities.[5-7] Triazole-thiadiazole fused compounds endowed with good anticancer activities and anti-inflammatory activity [8-10].

From various literature studies, here we reported the preparation of potential biologically active compounds and their Metal complexes (scheme-1). Invitro Antifungal activities of synthesized compounds were screened.

Experimental

The entire chemicals used were of laboratory grade. 2-(4-amino-5-mercapto-4H-1,2,4-triazol-3-yl)-N-phenylbenzamide be was prepared.[11]

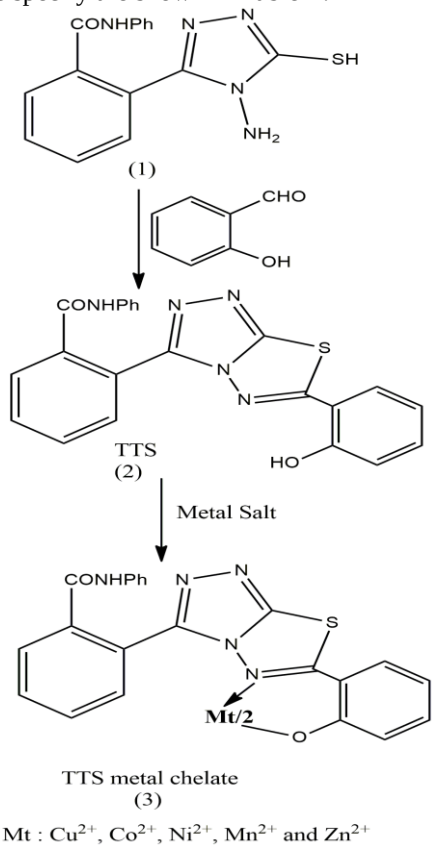
Synthesis of 2-(6-(2-hydroxy phenyl)-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-3-yl)-N-phenyl benzamide (TTS) (2):

2-(4-amino-5-mercapto-4H-1,2,4-triazol-3-yl)-N-phenyl benzamide (1) (0.01 mol) and salicyldehyde (0.01 mol) in dimethyl formamide (25 mL) were mix in presence of PTS was refluxed for 5-6 hrs, then cool it, cool and make it acidic. Filter, wash and recrystallized from EtOH. The yields was 76%, melting point was 194-195^{0}C.IR Spectral Features (cm^{-1})shows at 3350(OH),3328(NH),1652(CO), 1594,1580(C=N), 632(C-S-C),2850, 1630,1470 (aromatic C-H) and NMR Signals (δ ppm) at 7.01–8.12 (m,13H,ArH), 10.39(s,1H,NH), 5.40 (s,1H,OH).

Elemental analysis of TTS($C_{22}H_{15}N_5O_2S$):Calc.C% 63.91,N% 16.94,H% 3.66 and S% 7.76; Found C% 63.8, N%16.9,H% 3.6 and S% 7.7.

Preparation of TTS-metal-chelates:

In 150 ml solution of To a solution of EtOH-acetone and TTS (0.1 mole), we dropwise add 0.1N KOH solution, make it clear solution by water and diluted it to 250 ml. Now take 25 ml this solution and add drop wise to the solution of metal salt in H_2O. NaOAc or NH_3 was pour for fully precipitation, digest it on sand bath at 90° C for 2-2.5 hrs. Then filter, wash and dry it. The specify are shown in Table-1.



TTS metal chelate

(3)

Where Mt : Cu^{2+}, Co^{2+}, Ni^{2+}, Mn^{2+} and Zn^{2+}

Measurements

The elemental analysis were carried out by reported method [12]. Nicolet 760 FT-IR spectrometer and 60 MHz NMR spectrophotometer used for IR and NMR spectroscopic study. Magnetic susceptibility of the synthesized complexes was carried out. Antifungal analysis of prepared compounds was done by literature method[13].

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Table 1. Analysis of TTS Ligand and its Metal Chelates.

Comp.	% C	% H	%N	% S	% M
	Cald Found	Cald Found	Cald Found	Cald Found	Cald Found
TTS	63.91 63.8	3.66 3.6	16.94 16.9	7.76 7.7	- -
$TTSCu^{2+}2H_2O$ (923.54)	57.17 57.1	3.46 3.4	15.16 15.1	6.93 6.9	6.88 6.8
$TTSCo^{2+}2H_2O$ (918.94)	57.46 57.4	3.48 3.4	15.23 15.2	6.96 6.9	6.41 6.4
$TTSNi^{2+}2H_2O$ (918.71)	57.47 57.4	3.48 3.4	15.24 15.2	6.97 6.9	6.39 6.3
$TTSMn^{2+}2H_2O$ (914.94)	57.71 57.7	3.50 3.4	15.30 15.2	6.99 6.9	6.00 5.9
$TTSZn^{2+}2H_2O$ (925.38)	57.06 57.0	3.46 3.4	15.13 15.1	6.92 6.9	7.07 7.0

Results and Discussions

The synthesis of 2-(6-(2-hydroxyphenyl)-[1,2,4] triazolo [3,4-b][1,3,4]thiadiazol-3-yl)-N-phenylbenzamide(3) was yielded by reaction of 2-(4-amino-5-mercapto-4H-1,2,4-triazo l-3-yl)-N-phenyl benzamide(2)with salicyldehyde. The TTS elemental analysis (Table-1) are reliable with the structure expected (Scheme-1).

Table 2. TTS Metal Chelates: Spectral features and magnetic moment

Metal Chelates	μ_{eff} (BM)	Electronic spectral data (cm^{-1})	Transition
TTS-Cu^{2+}	2.53	23454 13216	Charge transfer $^2B_{1g}\rightarrow{}^2A_{1g}$
TTS-Ni^{2+}	3.70	22599 15373	$^3A_{1g}\rightarrow{}^3T_{1g}(P)$ $^3A_{1g}\rightarrow{}^3T_{1g}(F)$
TTS-Co^{2+}	4.75	23736 19106 8927	$^4T_{1g}(F)$ $\rightarrow{}^4T_{2g}(F)$ $^4T_{1g}(F)\rightarrow{}^4T_{2g}$ $^4T_{1g}(F)$ $\rightarrow{}^4T_{2g}(P)$
TTS-Mn^{2+}	5.53	23238 19036 16844	$^6A_{1g}\rightarrow{}^6A_{2g}\,{}^4E_g$ $^6A_{1g}\rightarrow{}^4T_{2g}$ (4G) $^6A_{1g}$ $\rightarrow{}^4T_{1g}(PG)$
TTS-Zn^{2+}	Diamag.	-	-

The 3350 cm^{-1} broad band shows hydroxyl group. NMR spectrum of TTS shows singlet for NH at 10.39 and 5.40 δ ppm for hydroxyl.The elemental analysis are reliable with the expected structure. Here we seen M:L ratio is 1:2.

Table 3. Antifungal Screening of TTS and its Metal Chelates.

Comp.	At 1000 ppm fungus inhibition(%)			
	AN	*BT*	*NS*	*FO*
TTS	69	70	72	74
TTS-Cu^{2+}	91	85	90	87
TTS-Zn^{2+}	80	83	89	88
TTS-Ni^{2+}	86	82	80	85
TTS-Co^{2+}	88	84	85	79
TTS-Mn^{2+}	85	81	89	80

Where, AN-Aspergillus Niger, BT-Botrydepladia Thiobromine, NS-Nigrospora Sp. and FO-Fusarium Oxyporium

The absence of hydroxyl group band indicates the formation of metalocyclic compound. Table-2 shows important IR data.

Metal chelates magnetic moments and diffuse electronic spectrum also seen in Table-2. The experimental μ_{eff} in the range 2.53-5.53 B.M are which are reliable with expected structure[14].

The screening of antifungal activity of TTS ligand and its all chelates (Table-3) metal chelates are high toxic than ligand.

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