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Synthesis, Characterization, and Antibacterial Study of Cadmium (II) Thiosemicarbazone Complexes

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Abstract: *The reaction of CdI_2 with thiosemicarbazone-based ligands such as $(benztscz H)_2$ and $(benzoptscz H)_2$ resulted inCdI_2 $(benztscz H)_2$ and CdI2$(benzoptscz H)_2$ complexes where benztscz H and benzoptscz H are benzaldehyde and benzophenone thiosemicarbazone respectively. The above complexes have been prepared and characterized by elemental analysis, infrared and nuclear magnetic resonance (^1H and $^{13}C\{^1H\}$) spectroscopic techniques. These complexes have been studied for antibacterial activities using Escherichia coli (gram-negative) and Staphylococcusaureus(gram-positive).The minimum inhibitory concentration response of both the complexes was found to be 400ppm against the strains.*

Keywords: Cd(II) Complexes; Cadmium Iodide, Thiosemicarbazone, Antibacterial Study

I. INTRODUCTION

Nowadays, the number of infections is caused by bacteria, parasites, viruses, and fungi because of the decreased inability of humans for anti-microbial resistance. The main reason for this is the genetic changes that occur in the pathogens when exposed to antibacterial drugs which result in the evolution of pathogen-resistant agents[1].Thiosemicarbazones and their metal complexes are being studied due to their wide range of biological properties[2]. The influence on the biological activity of the metal complexes can be brought about by the nature of the metal ion, its oxidation state, and the number and type of ligands bound to improve the potentiality of the drugs[3].The metal complexes of thiosemicarbazones are frequently more active than the free ligand or sometimes act as a transporter for activation of the ligand as the cytotoxic agent[4]. Thiosemicarbazones complexes possess awidespread set of N, S donor ligands which have shown their importance in coordination chemistry. These ligands became popular due to their great flexibility, which is exhibited by their ability to exist as two forms (thione-thiol), and the capacity to bind metal ions in the anionic or neutral form, working as a monodentate or bidentate ligand[5,6,7,8]. After the formation of complexes, there is delocalization of positive charge of the metal with the donor groups and in chelates which increases the hydrophobic nature which enhances penetration through lipid layers of microorganism causing the death of the bacterias[9]. The biological activity of thiosemicarbazones is dependent on their chelating ability with transition metal ions, bonding through N, N, and S atoms or O, N, and S atoms[10]. Various biological properties are demonstrated by the thiosemicarbazone complexes such as antibacterial, antifungal, antibacterial, antitumor, antioxidant, and DNA-interactions[11].

Various thiosemicarbazone complexes have been studied for the antibacterial activity on *E. coli* and *S.aureus*. The thiosemicarbazone complexes of metals include Pd(II) and Zn(II)[2], Cu(II)[12], 4- and 6- coordinate Ni(II)[13], Mn(II) and Cr(II)[10], Ag(I)[14], Co(II)[15], Cd(II)[3] and many more have been reported so far for the antibacterial activity.

In the present paper, we havesynthesized Cd(II) complexes [$CdI_2(benztscz H)_2$ and $CdI_2(benzoptscz H)_2$] of thiosemicarbazone ligands of the type the $CdI_2(LH)_2$.Here, benztscz H benzoptscz H are benzaldehyde and benzophenone thiosemicarbazone ligands. The synthesized complexes were used for the antibacterial activity studies of the bacterial species such as gram-positive (*Staphylococcus aureus*) and gram-negative (*Escherichia coli*) bacteria.

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II. EXPERIMENTAL

2.1 Synthesis of CdI_2 (benztsczH)$_2$

CdI2(benztsczH)2complex was reported in our previous work[16].In brief, CdI_2 (0.500 g) andbenzaldehyde thiosemicarbazone (0.5056 g) was mixed in 15 mL methanol each and refluxed for 2 h in RB flask. The solid obtained from clear solution was kept for crystallization. Melting Point: 206 °C, (Yield: 86.19 %), Elemental analysis (%) found (calculated): Cd: 20.60 (20.74), C: 35.35 (35.46), H: 3.29 (3.34), N: 15.70 (15.51), S: 11.69 (11.83), Cl: 13.02 (13.08). I.R. (cm-1): 3431, 3271 (ν_{NH2} asym and sym), 3190 (ν_{NH}), 1575 ($\nu_{C=N}$), 848 ($\nu_{C=S}$). N.M.R. (δ in ppm): 1H: 7.36-8.19 (s, 2H, -NH2; + m, 6H, C6H5-CH=); 11.42 (s, 1H, -NH-); 13C (δ in ppm): 178.23 (>C=S), 142.92 (>C=N-), 134.57, 130.32, 129.10, 127.76 (aromatic carbons)

$$CdI_2 + 2\,LH \xrightarrow{\text{Methanol, Reflux}} CdI_2(LH)_2 \qquad \text{--- (1)}$$

2.2 Synthesis of CdI_2 (benzoptsczH)$_2$

CdI_2(benzoptsczH)$_2$ complex was synthesized using the reported method[17]. In brief, 0.500 g of CdI_2 and 0.3486g benzaldehyde thiosemicarbazone was mixed into 15 mL methanol each. The above reactants are refluxed for 2 h in the RB flask. The solid obtained from the clear solution was kept for crystallization. Melting point: 91°C,yield: 1.572 g (97.88%), Elemental analysis for $CdI_2C_{28}H_{26}N_6S_2$, the calculated %: Cd, 12.81; C, 38.35; H, 2.98; N, 9.58; S, 7.31. and found%: Cd, 12.97; C, 38.54; H, 2.84; N, 9.28; S, 7.14. IR, cm–1: 3406, 3286 (ν_{NH2} asym and sym), 3192 ($\nu_{N–H}$), 1592 ($\nu_{C–N}$), 832 ($\nu_{C–S}$). 1 H NMR (δ, ppm): 7.32–8.48 (s, NH2, mC6H5– C=N), 8.63 (s, NH); 13C NMR (δ, ppm): 177.64 (C=S), 149.03 (C=N), 136.26, 131.17, 129.96, 129.77, 128.31, 128.25, 127.56 (aromatic carbons).

2.3 Preparation of Inoculums

Stock cultures were maintained at 4°C on slants of nutrient agar. Active cultures of the experiment were prepared by transferring a loopful of cells from the stock cultures to a test tube of Muller-Hinton broth (MHB) for bacteria that were incubated without agitation for 24 hours at 37°C. The cultures were diluted with fresh Muller-Hinton broth.

2.4 Materials

Cadmium iodide [CdI_2], benzaldehyde [C_7H_6O], benzophenone [$C_{13}H_{10}O$],and thiosemicarbazide [CH_5N_3S], were purchased from Sigma Aldrich, methanol [CH_3OH] was purchased from S. D. Fine Chem Ltd. Distilled water for the required reaction was distilled out in the laboratory. Muller-Hinton broth were purchased from S. D. Fine Chem Ltd. All the chemicals used in the synthesis of the complex were analytical grade and used without any purification.

2.5 Instrumentation

Elemental analyses were performed on Thermo Electron Corporation FLASH EA 1112 series analyzer. The infrared spectra (IR) were recorded as KBr pellets using KBr press on a Perkin Elmer FT-IR spectrometer in the 4000-400 cm^{-1} range. ^1H and $^{13}C\{^1H\}$ nuclear magnetic resonance (NMR) were recorded in a 5 mm NMR tube in DMSO-d_6 on a Bruker Avance II 300 MHz NMR spectrometer operating at 300 and 75.47 MHz, respectively. The chemical shifts were referred to as internal standard tetramethylsilane (TMS) for ^1H and $^{13}C\{^1H\}$ NMR.

III. RESULTS AND DISCUSSION

3.1 Antibacterial Susceptibility Test

The agar cup method was used to screen the antibacterial activity. In vitro antibacterial activity was screened by using Muller Hinton Agar (MHA). To the sterile molten MH agar butts of 20 ml, 0.1 microbial culture was added and poured into sterile Petri plates. The plates were allowed to solidify for 5 minutes. The wells were prepared using a cork-borer, the sample of different concentrations was added in wells and the plates were kept for incubation at 37°C for 24 hrs. At the end of incubation, inhibition zones formed around the wells were measured with a transparent ruler in millimeters. Additions are performed according to the addition table. The images for the zone of inhibition are given in figure 1.

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The minimum inhibitory concentration of $CdI_2(benztsczH)_2$ and$CdI_2(benzoptsczH)_2$ was found to be 400ppm against both test organisms *E. coli* and *S. aureus*(Table 1). Table 2 and 3 represents the measurement of the zone of inhibition for $CdI_2(benztsczH)_2$ and $CdI_2(benzoptsczH)$.

Table 1: Addition table for MIC

Conc. (in ppm)	Stock Sol. (ml)	Diluent (NB) (ml)	Total volume (ml)	Culture (ml)	$CdI_2(benztsczH)_2$		$CdI_2(benzoptsczH)_2$	
					Growth of *E. coli*	Growth of *S. aureus*	Growth of *E. coli*	Growth of *S. aureus*
100	0.5	4.5	5.0	0.1	++	++	++	++
200	1.0	4.0			+	+	+	+
300	1.5	3.5			+	+	+	+
400	2.0	3.0			-	-	-	-
500	2.5	2.5			-	-	-	-
600	3.0	2.0			-	-	-	-
700	3.5	1.5			-	-	-	-
800	4.0	1.0			-	-	-	-
900	4.5	0.5			-	-	-	-
1000	5.0	-			-	-	-	-

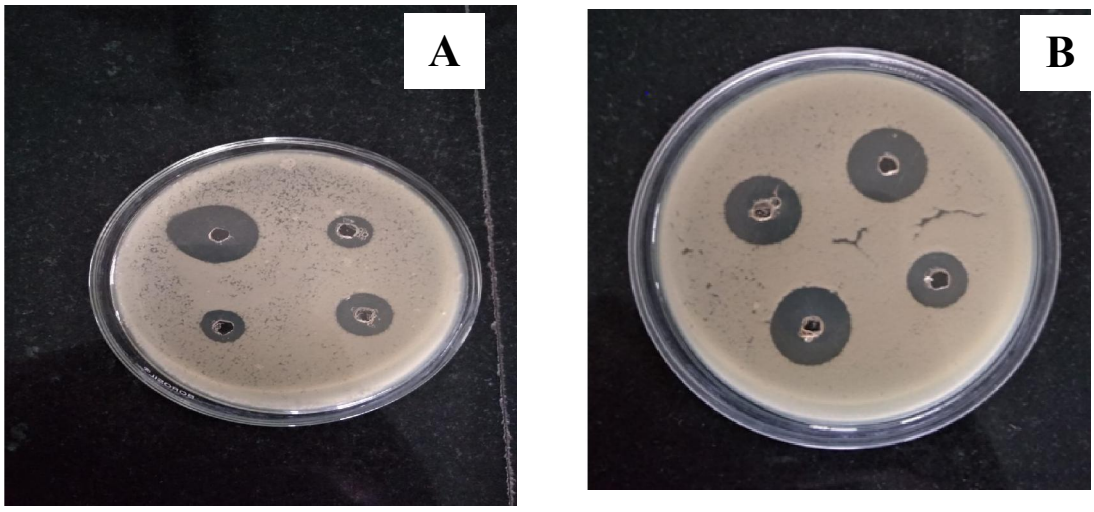

Figure 1: A representative antibacterial activity of $CdI_2(benztsczH)_2$with test organism (A) *E. coli* and (B) *S. aureus*

Table 2: Measurement of zone of inhibition for $CdI_2(benztsczH)_2$

Conc. of $CdI_2(benztsczH)_2$ (in ppm)	Diameter (in mm) for $CdI_2(benztsczH)_2$ for *E. coli*	Diameter (in mm) for $CdI_2(benztsczH)_2$*for S. aureus*
200ppm	11mm	12mm
300ppm	12mm	15mm
400ppm	15mm	18mm
500ppm	20mm	20mm

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Table 3: Measurement of the zone of inhibition for $CdI_2(benztsczH)_2$

Conc. of $CdI_2(benzoptsczH)_2$ (in ppm)	Diameter (in mm) for $CdI_2(benzoptsczH)_2$ for *E. coli*	Diameter (in mm) for $CdI_2(benzoptsczH)_2$ *for S. aureus*
200ppm	10 mm	13 mm
300ppm	13 mm	16 mm
400ppm	15 mm	17 mm
500ppm	18 mm	19 mm

IV. CONCLUSION

The antibacterial activity of $CdI_2(benztsczH)_2$ and $CdI_2(benzoptsczH)_2$ have been tested against *E. coli* and *S. aureus* for minimum inhibitory concentration and zone of inhibition. The minimum inhibitory concentration was found to be 400ppm against both test organisms. The complexes of $CdI_2(benztsczH)_2$ and $CdI_2(benzoptsczH)_2$ were found to be effective antibacterial agents against *E. coli* and *S. aureus*.

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