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Research Article

Spectroscopic characterization, and anti-bacterial activity studies of the benzohydrazide derivatives complexes

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Abstract

Five bivalent metal complexes of Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) with N-(4-(dimethylamino)benzylidene)benzohydrazide (HDmby) ligand with the general formula $[M(Dmby)_2(H_2O)_2]$ {where M^{II}= Co(1), Ni(2) and Cu(3)} and $[M(Dmby)_2]$ {where MII= Zn(4), and Cd(5)} have been synthesized and characterized. The prepared complexes were characterized by ^1H NMR, IR spectrometry, UV–Vis spectra, Conductivity measurements, magnetic susceptibility. The Co(II), Ni(II) and Cu(II), complexes were found to have octahedral geometry, whereas the Zn(II) and Cd(II) complexes were of tetrahedral geometry. The anti-microbial activity of the synthesized complexes were investigated against three pathogenic bacteria using inhibition zone method and the order of activity of the complexes was determined to be $[Zn(Dmby)_2]$ > $[Cu(Dmby)_2(H_2O)_2]$ > $[Ni(Dmby)_2(H_2O)_2]$ > $[Co(Dmby)_2(H_2O)_2]$ > and $[Cd(Dmby)_2]$.

Keywords: Benzohydrazide, Bivalent Metal, Complexes, Anti-Bacterial and Anti-Fungi

Introduction

Benzohydrazide derivative complexes are coordination compounds that coordinate benzohydrazide derivative ligands to a central metal ion or atom. Benzohydrazide derivative ligands contain both a hydrazine group (-NH-NH_2) and a benzoyl group (-$C_6H_5C(O)$-). The coordination of these ligands with a metal center results in a complex with unique properties and potential applications (Shakdofa et al., 2014; Kumar et al., 2017). Benzohydrazide derivative ligands and their complexes can vary depending on factors such as the choice of metal ion, the nature of the ligands, and the overall structure of the complex. Researchers continue to explore and investigate these complexes' properties and potential applications in various scientific fields.

These complexes can have a range of applications. Benzohydrazide derivative complexes can act as catalysts in various chemical reactions, influencing the catalytic activity and selectivity, making these complexes valuable in synthetic chemistry (Tabernero et al., 2002; Pelagatti et al., 2003; Chen et al., 2003), which has also been studied for their potential pharmacological activities. They might exhibit antibacterial, antifungal, antitumor, or anti-inflammatory properties. Researchers explore their interactions with biomolecules for potential drug development. It can also be used to understand the interaction of benzohydrazide complexes with biologically relevant molecules and could provide insights into their potential role in biological systems (Zhong et al., 2007; ElTabl et al., 2015; Okagu et al., 2019; Katouah et al., 2019; Al-Qadsy et al., 2021; Korkmaz et al., 2022). In addition, it can contribute to developing new materials with specific properties. These materials can be used in sensors, electronics, and optics. Moreover, benzohydrazide derivative complexes can participate in the formation of coordination polymers, which are extended networks of metal-ligand interactions. These polymers could have applications in gas storage, molecular sieving, and other areas (Zuo et al., 2012; Burgos-Lopez et al., 2019; Theppitak et al., 2021; Guan et al., 2022; Zhao et al., 2022; Sánchez-Fernández, 2023). Herein, the synthesis, spectroscopic studies, as well as the anti-bacterial

studies for N-(4-(dimethylamino) benzylidene) benzohydrazide ligand has been reported.

Experimental part

General

The chemical compounds and solvent were provided and used without purification. UV–visible spectra are measured within 900–200 nm using Cary 100 spectrophotometer in DMSO solutions. The FT-IR spectra were measured in 400 –4000 cm^{-1} as KBr disc on a SHIMADZUFT-IR apparatus. The molar conductance was recorded in DMSO solution at 10^{-3} M at 25 °C using Oakton EC Tester 11 dual-range, conductivity tester. The NMR spectra were specified on Bruker 400 MHz spectrometer using DMSO-d_6 as a solvent. The magnetic susceptibility was determined by Sherwood magnetic susceptibility balance at room temperature.

Preparation of the complex ($Co(Dmby)_2(H_2O)_2$) (1)

A hot solution of the cobalt chloride hexahydrate (0.05g, 0.211mmol) in distal water (10mL) was added to a mixture of an ethanolic solution of HDmby ligand (0.113g, 0.422mmol) and sodium hydroxide (0.017g, 0.422mmol) in (15mL). A blue ppt. was produced. The mixture was stirred at 3h. The brownish blue ppt. produced was filtered off washed with distal water and ethanol and dried under vacuum.

The following complexes $[Co(Dmby)_2(H_2O)_2]$ (2), $(Co(Dmby)_2(H_2O)_2]$ (3), $[Zn(Dmby)_2]$ (4) and $[Cd(Dmby)_2]$ (5) were prepared and isolated in similar method.

Anti-microbial studies

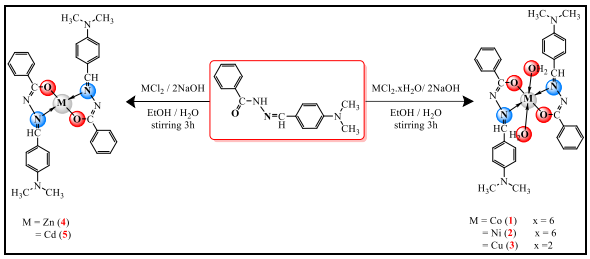
The antibacterial activity of synthesized complexes of N-(4-(dimethylamino) benzylidene) benzohydrazide ligands were carried out on *Bacillus subtilis*, *Staphylococcus aureus*, and *Escherichia coli* by using diameter inhibition zone method. Tetracycline was used as a standard drug at 10^{-3} M in DMSO. The standard error for the experiment was ± 0.03 %, and the experiments were repeated three times under the same conditions.

Results and Discussion

Synthesis

The reaction of two moles of two equivalent molar of N-(4-(dimethylamino)benzylidene)benzohydrazide (HDmby) ligand with bivalent metal chlorides of Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) (Scheme 1), afforded complexes of the formula $[M(Dmby)_2(H_2O)_2]$ {M^{II}=Co(1), Ni(2) and Cu(3)} and $[M(Dmby)_2]$ {where M^{II}=Zn(4), and Cd(5)}. The results indicated that the HDmby ligand coordinated as bi-dentate chelated mode via the nitrogen atoms of azomethine and oxygen atom of carbonyl group to afford octahedral around Co(1), Ni(2) and Cu(3) ions or a tetrahedral shape a round Zn(4), and Cd(5). The prepared complexes were described by ^{1}H NMR, IR spectrometry, UV–Vis spectra, conductivity measurements, magnetic susceptibility (Table 1-3).

Scheme 1. Preparation of complexes (1- 5)



The conductivity measurements of the prepared complexes showed that complexes have a low conductivity value and fell within the range of non-electrolytic complexes (Table 1). Also, the CHN analysis of the prepared complexes was measured and the results showed that the obtained experimental values agree with theoretical values, which is consistent with the proposed formulas for the prepared complexes (Table 1).

Table 1. Color, molar conductivity, m.p(°C), yield (%) and CHN analysis

No.	Complexes	Color	Λ*	m.p(C°)**	Yield %	CHN analysis (%) Calc. (found)		
						C	H	N
1	$[Co(Dmby)_2(H_2O)_2]$	Green	12.1	300d	71	61.24 (61.43)	5.78 (5.69)	13.91 (14.09)
2	$[Ni(Dmby)_2(H_2O)_2]$	Yellowish green	11.8	283-285	73	61.26 (62.36)	5.78 (5.89)	13.40 (13.55)
3	$[Cu(Dmby)_2(H_2O)_2]$	Brownish blue	6.7	190-192	77	60.79 (60.91)	5.74 (5.82)	13.29 (13.42)
4	$[Zn(Dmby)_2]$	White	3.6	300-303d	59	64.27 (64.40)	5.39 (5.42)	14.05 (14.21)
5	$[Cd(Dmby)_2]$	Light yellow	4.0	198-199	51	59.58 (59.61)	5.00 (4.93)	13.03 (13.23)

* Molar conductivity was measured in DMSO solvent at 10^{-3} M (ohm^{-1}. cm^2. mol^{-1})
** d = decomposed

IR spectra data

Infrared technology is one of the techniques that are used in the characterized of chemical compounds, especially the functional groups in chemical compounds such as ν(C=O), ν(C=N), ν(C=S), ν(N-H), and ν(N-N), groups…etc.

The IR spectra of the prepared complexes (1-5) shown in Fig. 1 and 2 displayed a strong to medium band within the range (1638-1652) cm^{-1}, due to the frequency of the carbonyl group ν(C=O), which shifted towards lower frequencies (within the range of 25-38 cm^{-1}) (in free ligand 1676cm^{-1}), and this indicates that the ligand (HDmby) coordinates through the oxygen of the carbonyl group (Zhong et al., 2007; Shakdofa et al., 2014; Salih et al., 2022; Al-Janabi et al., 2023; Afandi et al., 2023; Al-Janabi et al., 2023). Also the spectra also showed a strong band within the range (1553-1581) cm^{-1} due to the frequency of the azomethine group ν(C=N), which shifted towards lower frequencies, and this indicates that the ligand coordinates through the nitrogen atom of the azomethine group (Okagu et al., 2019; Al-Janabi et al., 2022; Mohamed et al., 2023) This reinforces the proposed formula for the complexes and the fact that the ligand (HDmby) was coordinated in a bidentate chelated ligand with nitrogen and oxygen atoms. Also, the spectra showed a broad band within the range (3398-3421) cm^{-1}, which is due to of the ν (H_2O) (Okagu et al., 2019; Al-Janabi et al., 2021; Al-Jibori et al., 2021). In addition, the IR spectra of complexes (1-3) showed two bands ρr (H_2O) within (839-871 cm^{-1}) range and ρw (H_2O) at (641-650 cm^{-1}) range which designate the presence of coordinated water molecules(Rosi & Bauschlicher, 1989; Waheeb et al., 2022). The other bands are listed in Table 2.

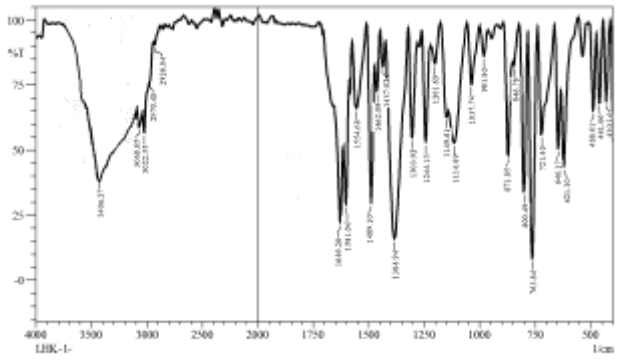


Fig. 1 IR spectrum of the complex [Co(Dmby)$_2$(H_2O)$_2$]

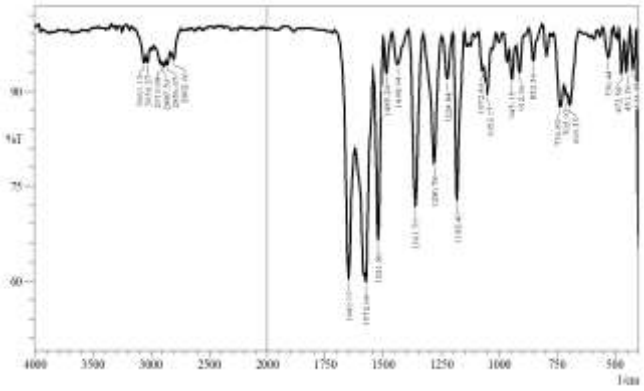


Fig. 2 IR spectrum of the complex [Cd(Dmby)$_2$]

Table 2. IR selected data (in cm^{-1}) of the prepared complexes 3.3 ^{1}H-NMR data of complexes (Zn(Dmby)$_2$) and (Zn(Dmby)$_2$)

No.	Complexes	ν(H_2O)	ν(CH) Ar. Aliph	ν(C=O)	ν(C=N)	ν(C=C)	ρr(H_2O) ρw(H_2O)	νM-O νM-N
1	[Co(Dmby)$_2$(H_2O)$_2$]	3406b	3068w 2970w	1646s	1581s	1554s	871s 646s	461m 430m
2	[Ni(Dmby)$_2$(H_2O)$_2$]	3398b	3058w 2831w	1641s	1572s	1552s	839s 650s	459m 432m
3	[Cu(Dmby)$_2$(H_2O)$_2$]	3421b	3051w 2970w	1652s	1553s	1542s	862s 641s	473m 421m
4	[Zn(Dmby)$_2$]	-	3061w 2910w	1638s	1569s	1542s	-	461m 430m
5	[Cd(Dmby)$_2$]	-	3068w 2970w	1649s	1572s	1521s	-	461m 430m

The ^{1}H-NMR spectra of complexes [Zn(Dmby)$_2$] (4) and [Cd(Dmby)$_2$] (5) (Fig. 3). The spectra displayed the protons of azomethine group as a singlet peak at δH = 8.37ppm and 8.33ppm, for the two complexes respectively. Also a singlet peak at δH = 2.96ppm and δH = 2.98ppm, due to methyl groups, respectively. The aromatic protons displayed as two doublet peaks and one multiplet peaks. The first doublet peak showed at δH = 7.86ppm (J_{HH} = 7.46Hz) and δH= 7.90ppm (J_{HH} = 8.00Hz). The second doublet peak showed at δH = 6.74ppm (J_{HH} = 8.43Hz) and δH = 6.77ppm (J_{HH} = 8.00Hz), the integration of each signal indicates that it corresponds to four protons. Finally, the multiplet peak showed at δH = 7.51

ppm and δH = 7.56 ppm, for the two complex, respectively and their integration indicates that they correspond to ten protons.

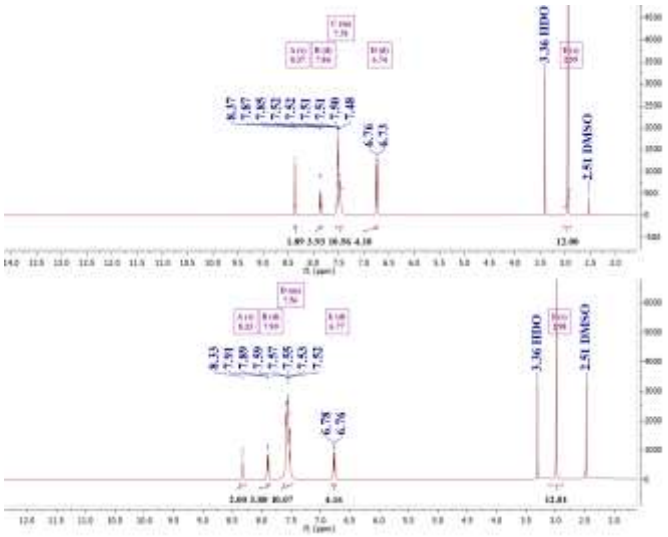


Fig. 3 ^{1}H-NMR spectra of ($Zn(Dmby)_2$) (above) and ($Zn(Dmby)_2$) (down)

UV-Visible spectra

The Uv-visible spectra of the complexes $[Co(Dmby)_2(H_2O)_2]$, $[Ni(Dmby)_2(H_2O)_2]$, and $[Cu(Dmby)_2(H_2O)_2]$ was measured in DMSO solution.

The $[Co(Dmby)_2(H_2O)_2]$ showed the μeff value of 4.83B.M. which due to three odd electrons. The spectrum of the $[Co(Dmby)_2(H_2O)_2]$ complex showed a bands at 263, 320, 471 nm which assigned to $^4T_1g \rightarrow {}^4T_2g$, $^4T_1g \rightarrow {}^4A_2g$, $^4T1g \rightarrow {}^4T_1g(p)$, respectively, this refer to octahedral complexes of cobalt(II) (Rosi & Bauschlicher, 1989; Waheeb et al., 2022; Salih et al., 2022; Al-Janabi et al., 2023).

The $[Ni(Dmby)_2(H_2O)_2]$ displayed the μeff value of 3.17 B.M. which due to two unpaired electrons. The spectrum of the $(Ni(Dmby)_2(H_2O)_2)$ showed four peaks at 334, 391, 450, 660 nm, due to $^4T_1g \rightarrow {}^4T_2g$, $^4T_1g \rightarrow {}^4A_2g$, $^4T_1g \rightarrow {}^4T_1g_{(F)}$ and LMCT respectively, this due to octahedral complexes of Ni(II) (Rosi & Bauschlicher, 1989; Waheeb et al., 2022; Salih et al., 2022; Al-Janabi et al., 2023).

The $[Cu(Dmby)_2(H_2O)_2]$ displayed the μeff value of 1.81 B.M. which due to one odd electron. The electronic spectrum of the Cu(II) complex showed two peaks at 345 and 405 nm, due to LMCT, Also the spectrum displayed a peak at 628 nm, which due to the d-d electronic transition type $^2Eg \rightarrow {}^2T_2g$ transition, this due to distorted octahedral complex of Cu(II) (Rosi & Bauschlicher, 1989; Waheeb et al., 2022; Salih et al., 2022; Al-Janabi et al., 2023). The $[Zn(Dmby)_2]$ and $[Cd(Dmby)_2]$ are diamagnetic and the Uv-visible spectra displayed the peaks of the ligand and LMCT 278 and 330 nm.

Antibacterial Activity

The antibacterial activity of the prepared compounds and control drugs were examined against three pathogenic bacteria (*Bacillus subtilis*, *Staphylococcus aureus*, and *Escherichia coli*) using inhibition describe by Bauer et al 1959. The activities of the compounds were compared with Tetracycline. The dimeter inhibition zone result are listed in Table 3 and Fig 4.

In general, the metal complexes displayed good inhibitory effect when compared to the ligand against all tested bacteria strains with zone inhibition in the range of 19-34 mm. The compound with Cu- complexes showed highest antibacterial activities with the zone inhibition diameter ranging from 23 to 34 mm against all tested bacteria strains. This phenomenon can be explained based on Tweedy's chelation (21, 22). The chelation reduces the polarity of metal cation to some extent due to the overlap of the ligand orbital and partial sharing of the positive charge of the metal ion. The chelation increases the delocalization of p-electrons over the whole chelate ring and enhances the lipophilicity of the complexes which, in turn, increases the penetration of the complexes into lipid membranes, and results in blockage of metal sites in the enzymes of the microorganisms. In addition, metal complexes hinder the respiration process of the cell and, block the synthesis of proteins and prevent further growth of the organism.

Table 3. DIZ (mm) of the prepared complexes against three pathogenic bacteria

Complexes	DIZ (mm)		
	Bacillus subtilis	***S. aureus***	***E. coli***
$[Co(Dmby)_2(H_2O)_2]$	15	19	20
$[Ni(Dmby)_2(H_2O)_2]$	22	18	16
$[Cu(Dmby)_2(H_2O)_2]$	20	18	21
$[Zn(Dmby)_2]$	22	25	23
$[Cd(Dmby)_2]$	14	17	15
Tetracycline	24	26	31

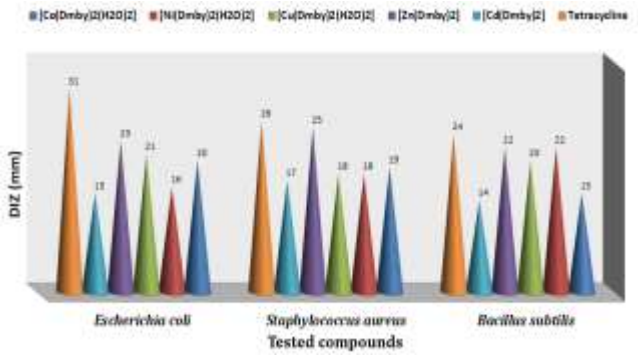


Fig. 4 Represent the biological activity of the prepared complexes against three pathogenic bacteria

Conclusion

Treatment of the N-(4-(dimethylamino)benzylidene) benzohydrazide with metal salt chlorides afforded complexes of the types $[M(Dmby)_2(H_2O)_2]$ {where M^{II}= Co(1), Ni(2) and Cu(3)} and $[M(Dmby)_2]$ {where M^{II}= Zn(4), and Cd(5)}. These complexes were characterized by different spectroscopic and physical methods. The Co(II), Ni(II) and Cu(II), complexes have octahedral shape, whereas the Zn(II) and Cd(II) complexes have tetrahedral geometry. The anti-microbial activity of the synthesized complexes was investigated against three pathogenic bacteria using inhibition zone method and the order of activity of the complexes was determined to be $[Zn(Dmby)_2]$ > $[Cu(Dmby)_2(H_2O)_2]$ > $[Ni(Dmby)_2(H_2O)_2]$ > $[Co(Dmby)_2(H_2O)_2]$ > and $[Cd(Dmby)_2]$.

Conflict of Interest

The author hereby declares no conflict of interest.

Consent for publication

The author declares that the work has consent for publication.

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