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Synthesis, characterization and antibacterial studies of 2-hydroxyacetophenone Nickel (II) complex

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Abstract

A ternary metal complexes of Ni(II) where HL representso-hydroxyacetophenone as a ligandwas synthesizedin this work. The coordination behavior of HL towards the selected metal ions in addition todetermination of the geometry of complexes have been deduced from the elemental analyses, spectral techniques (IR, and UV–Vis), conductivity and magnetic susceptibility measurements.Correlation of all spectroscopic and analytical data reflect that; HL behaves as a neutral bidentate ligand,octahedral geometry are assigned for Ni(II) complex. The thermal stability of the metal complex in comparison to HL was investigated through the thermal studies , also the number and the nature of the water molecules was identifiedby thermal study. HL and its complex was screened for in vitroantimicrobial activities against a set of bacterial and fungal species using agar well diffusion method.

Key words: *Complexes, thermal studies,antimicrobial activity.*

Introduction

Acetophenone and its substituted analogues are class of compounds that naturally occurring and found in many foods and plants. They exhibit interesting physicochemical and biological properties. Also, they have been used in cosmetics, perfumes and organic synthesizes well as a flavoring agent in some industries[1-3]. They belong to a class of organic compounds known as ketones because they have at least one carbonyl group. Additionally,2-hydroxyphenones are known to have strong coordinating properties and complexing ability towards the metal ions adopting several geometries[4-6].Transition metal ions with different oxidation state have a strong role in bioinorganic chemistry and redox enzyme systems and may provide the basis of models for active sites of biological systems. Transition metal complexes have significant important in designing metal-based drugs and they used in treatment many human and animals diseases as a result to their versatile spectroscopic properties and their promising pharmacological applications[7,8].An interesting property of transition metal complexes is that they can adopt a wide range of molecular shapes with various coordination modes including linear, tetrahedral, square planar, octahedral structures and other depending on the coordination number of the metal ion. This function are unavailable to purely organic compounds[9,10].Recently, many accomplishments have been performed in the materials, particles have attracted great interest in recent years because of their unique chemical and physical properties, which are different from those of either the bulk materials or single atoms [11].

Based on all the previous facts, the present work is focused on the synthesis of novel sized Ni(II), Cu(II) and Cd(I) complexes of 2-hydroxyacetophenon ligand(HL). The structures of Ni(II), Cu(II) and Cd(I) complexes were elucidated by elemental analysis, TGA, magnetic moment measurements, molar conductance, UV-Vis, X-ray powder diffraction, IR, SEM, TEM, 1 H-NMR, and mass spectral studies, in addition, The synthesized compounds were screened for in vitro antimicrobial activity and antioxidant properties.

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2. Experimental

2.1. Materials and physical measurements

The metal ions selected for metal complex preparationnamely, $NiCl_2.6H_2O$ was purchased from El Goumhoria Trade Pharmaceuticals & Chemicals Company, Cairo (Egypt), while *o*-hydroxyacetophenone, ethanol , diethyl ether and silver nitrate were analytical grade chemical obtained from Merck chemical company.. All materials and solvents were used without furtherpurification.

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The elemental analyses of carbon, hydrogen, nitrogen and sulphur were performed using a Perkin-Elmer CHN 2408 CHN elemental analyzer at Micro Analytical Center, Cairo University, Giza, Egypt.

The infrared spectra of the ligands and the isolated solid complexes were recorded using KBr discs on a Perkin-Elmer 437 IR spectrophotometer (400-4000 cm^{-1}) at Micro Analytical Center, Cairo University, Giza, Egypt.

2.2. Preparation of the metal complexes.

10 ml of a 0.01 M of the metal salts solution in EtOH were positioned in a high-density ultrasonic probe, operating at 24 kHz with a maximum power output of 400 W. Into this solution 10 ml of a 0.01 M solution of the ligands were added drop wise. The obtained precipitates were allowed to evaporate at roomtemperature to obtain metal complexes nanoparticle in a powder form.

2.2.1. Antimicrobial activity

To evaluate the antimicrobial activity of the investigated compounds; HL ligand and its metal complexes, a variety of bacterial and fungal species were chosen as follows:Four bacterial species ie, *Staphylococcus aureus* (RCMB010010) and *Bacillus subtilis* (RCMB015(1)NRRL B-543 (Gram-positive bacteria); *Escherichia coli* (RCMB 010052) ATCC 2599 and *Proteus vulgaris* RCMB (1) ATCC 13315(Gram-negative bacteria)in addition to two fungal strains, *Aspergillus flavus* (RCMB 002002) and *Candida albicans* RCMB 005003(1) ATCC.In addition,*Gentamycin*(4µg/mL)and *ketoconazoleas*(100µg/mL)were used as standard antibacterial and antifungal agents, respectively

In vitro antimicrobial activity studies were carried out using the agar well diffusion technique[12,13]. At first, the tested compounds were solubilized in DMSO, which does not have an inhibition activity, to get a final concentration of 10 mg mL^{-1}.The second step was that the nutrient agar (NA) and Sab. dextrose agar (SDA) were seeded with aliquots of the test bacteria and fungi species,respectively thenleft tocool and solidify.After that, wells (6 mm in diameter) weremade in the solidified agar using a cork borer and loaded with examined sample solutions. Finally, the agar plates are incubated under suitable conditions depending upon the test microorganism. The antimicrobial activity was indicated by the presence of clear inhibition zones around the wells as a result to diffusethe tested compounds in the agar medium and inhibits the growth of the microbial strain tested.

3. Results and discussion

The reactions of *o*-hydroxyacetophenonewith Ni(II) ions were prepared was. The results obtained were in good agreement with those calculated for the suggested formulae.All the metal complexes are coloured, solid and stable towards air and moisture at room temperature. The melting points were sharp, indicating the purity of the prepared metal complexes. The resulting metal complexes insoluble in water, carbon tetrachloride and chloroform but soluble in DMF and DMSO.

3.1. Elemental analysis

Microanalytical data obtained for the synthesized metal complexesconfirmed that the complexes are mononuclear, the ligand to metal ratio was found to be 1:1.The elemental analysis data were found to be in goodagreement with the proposed molecular formula, Table (1)

Table 1: Analytical and some physical data of the synthesized metal complexes

Metal complex	M.Wt.[(a)] Found(Calcd)	Elemental analysis, Found / (Calcd.) %				$\Lambda^{(c)}$
		C	H	Cl	M	
(1) $[Ni(HL)Cl_2].3H_2O$	(320.29) 319.83	29.71 (29.20)	3.90 (4.60)	22.31 (21.55)	18.03 (17.91)	4.44

3.2. Conductivity measurements

Molar conductivity measurements of 10^{-3}M in dimethyl sulfoxide solution (DMSO-d_6)of the synthesized metal complexes were recorded at room temperature and listed in Table 1. The values obtained were in the range 4.44-0.14 $\Omega^{-1}cm^2mol^{-1}$. These valuesconfirmed the nonelectrolytic nature of the metal complexes[14].

3.3. IR spectral studies and investigation of the ligand to metal binding modes.

IR spectra of Ni(II) complexwas compared with that of their parent ligand, HL to find out the points of attachment of the ligand to the metal ions in their complexes and assigning the coordination mode. The peak around 1643 cm^{-1} was assignedto υ(C=O) stretching mode [15].Thebands appearing in the region 1489, 1157 and 756 cm^{-1}were the usual modes of phenyl ring vibration[16].Upon coordination of the ligand, HL to the metal ions, there is a significant changes in the spectra of Ni(II), Cu(II) and Cd(II)complexes with respect to the free ligand spectrum, additionally, IR spectra of all complexes,Figure1exhibit similar features indicating the similarity of their molecular structures. In IR spectra of the complexes, the C=Ostretching frequency was observed in the region around 1663 – 1597 cm^{-1}. This shifting of υ(C=O) attributed to complex formation[17].

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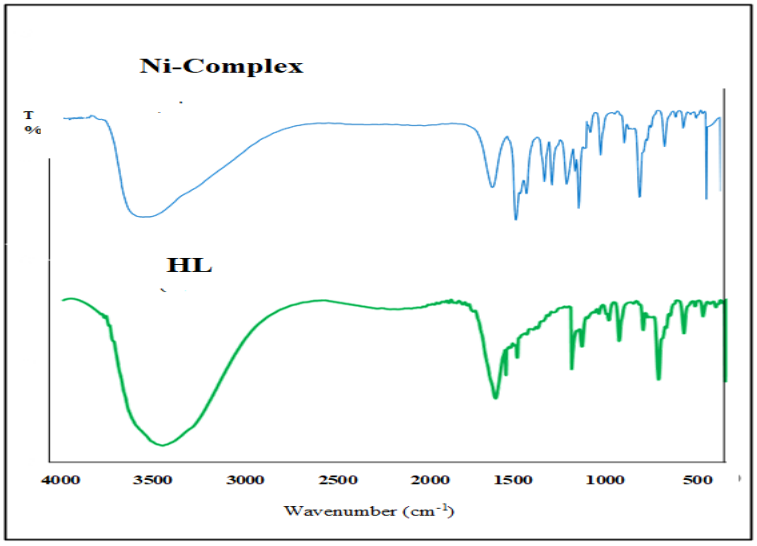

Figure (1): IR spectra ofHL ligand and its Ni complex

3.4. Magnetic and Electronic spectral studies

The magnetic behavior of Ni(II)complex was determined at room temperature. It appears from the magnetic susceptibility measurement that Ni(II) complex displayparamagnetic property and itsmagnetic moment value is 3.14BM indicating presence of two unpaired electrons andsuggesting octahedral geometry around Ni(II)ions[18].

The electronic reflectance spectra in the solid state of the free ligand, HL and its metal complexes are measured on the powder sample from 200 to 800 nm. The stereochemistry of the metal ions in thecomplexes can be assign via the electronic spectralmeasurements. The diffuse solid reflectance spectra of the ligand, HL showed three bands at 214, 273 and 365 nm. The higher energy band is assigned to Π - Π* transitionsofthe benzene &acetyl group. Additionally, Thelower energy band is attributed to charge transfer (CT) transitionswithin the molecule [19].

Upon interaction of the ligand with the selected metal ions and comparison of the spectrum of the free ligand with its Ni(II), complex, new bands were observed in the spectra of the complex at 550, for Ni complex due to LMCT which can be taken as a positive evidence of complex formation.

3.5. Thermogravimetric analysis.

From TG / DTGthermogram shown in Figure2, the mass loss in ligand as well as its complexes was calculatedfor the different steps of decomposition process and compared with those theoretically calculatedfor the suggested formulae. The temperature ranges, percentage mass losses, and the nature of each event of the decomposition reactions are given in table 2together with the decomposed and residual species. The important feature were obtained:

For Ni complex, the first step was in the temperature range of 55 – 141 ^{O}C, with the mass loss of 16.45 %(calc: 16.43%) which account for three lattice water molecules. The second and third steps occur within the temperature range 145 – 609 ^{O}C which corresponds to the removal of organic molecule with a mass loss of 26.75% (calc: 27.27%) and 33.11(calc: 32.60%) respectively . Metal oxide, NiO, was leaving as residue (found: 23.56 %, calc: 23.7%).

Empirical formula	Stage	Temp. range (^{o}C)	Weight loss%		Evolved moiety	Composition of the residue
			(Calc.)	Found		
[NiLCl$_2$]3H$_2$O [NiC$_8$H$_8$O$_2$Cl$_2$]. 3H$_2$O	I	125-215	(16.90)	17.52	3 H$_2$O hydrate	NiC$_8$H$_8$O$_2$Cl$_2$
	II	215-578	(35.32)	34.62	½O$_2$, Cl$_2$, CH≡CH	NiC$_6$H$_6$O
	III	674-856	(24.43)	24.51	3CH≡CH	NiO (23.35%)

Table (2): Thermo gravimetric analyses (TGA) results of the Nicomplex.

3.6. Biological activity

Antimicrobial activity of the isolated metal(II) complexes, were measured towards two grams positive bacteria species namely, *Streptococcus pneumonia* and *Bacillus subtilis*, two grams negative bacteria species, *Salmonella typhi* and *Escherichia coli* in addition to tow fungi species which are *Aspergillus fumigatus*, *Syncephalastrumracemosum*, The antibiotic, Gentamycin was used as standard antibacterial control and ketoconazole as standard Fungi control. The antibacterial

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and antifungal activities are tabulated in Table 3and have been indicated that:

- The results showed that the activity of the ligand became more pronounced when coordinated with the metal ions. The biological activity of the complexes follow the order: Antibacterial effect: Ni(II) > ligand.

Table 3: Antimicrobial assay of the free ligand HL and its metal complexes.

Tested organism	Compounds under study	
	HL	Ni
	Zones diameter showing complete growth inhibition in mm*	
Gram-positive bacteria:		
Staphylococcusaureus (RCMB010010)	16	14
Bacillus subtilis (RCMB015(1)NRRL B-543	18	14
Gram-negative bacteria:		
Proteus vulgaris RCMB (1) ATCC 13315	20	15
Escherichia coli (RCMB 010052)ATCC 2599	16	12
Fungi*:*		
Aspergillus flavus (RCMB 002002)	—	—

Conclusion

In the present research studies, our efforts were to synthesize and characterize Ni(II) complex. The synthesized compound was characterized by various physicochemical and spectral analysis. The acetophenone ligand binds with the metal ions in a bidentate manner, with O donor sites of phenolic-OH and C=O. Thermogravimetric studies reflect their thermal stability. The antimicrobial data showed that the metal complexes to be more biologically active compared to the ligand against all antibacterial species, the results also exhibit more activity for the Ni complex. Such studies may assist to search some novel chemotherapeutics to answer the emerging problem of drug resistance in health sciences.

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