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Synthesis and Spectral Analysis of Chromium, Copper Complexes of 2-Hydroxy-1, 2-diphenylethan-1

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Various chemical process has been utilized to analyze chromium (II) and copper (II) complexes with Benzoin. The fore mentioned complexes were analyzed for elemental analyses, and characterized by infrared, electronic spectra, NMR spectroscopy. The data analysis showed the metal ion formation of 1:1 ligand to metal ratio representing the [Cr (B)] H_2O and [Cu (B)] $2H_2O$ ionic complex in which B represents the Benzoin. According to the data analyzed by Infrared spectroscopy, it is through the carbonyl and hydroxyl oxygens of the Benzoin that chelation chemistry takes place. The complex structural analysis was elucidated by electronic spectral data analysis and by proton NMR spectroscopy. The complex systems with the metals also showed antimicrobial property against many bacterial strains.

Keywords: Benzoin, Coordination complex, Chromium, Copper, antimicrobial.

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Introduction

Benzoin also known as 2-hydroxy-1, 2-phenylethanone has diverse usages in the chemical and medical industries. It has been used in anti-inflammatory responses involving throat and larynx physiology, as well as inhalation and evaporation techniques utilized to sooth and relieve respiratory distress. Moreover, Benzoin has also been used in dermatological settings to heal skin fissures (1). It has been noted that Salicylaldehyde and Benzoin derived Co (II), Ni(II) and Cu(II) mixed ligand complexes were recognized to possess an octahedral geometry when analytical elemental analyses such as molar conductance measurements, infrared and electronic spectra were utilized. (2). Complexes of the type of ML_2X_2 and ML_2, with M as Cr(III), Cu(II) or Zn(II) ion and benzointhiosemicarbazone as L, with nitrate or chloride as X were characterized by conductance measurements, IR and electronic spectra(4). Likewise, Chromium (III) α-benzoin oxime complexes were generated and analysed by elemental analyses, infrared, electronic absorption and ^{1}HNMR spectra (5). This paper will further expand on the formation of benzoin ligand metal complex interactions formed between benzoin and manganese (II). This study will examine the creation and clarification of intricate geometrical structures (6). Due to its layered structure, the two-dimensional transition metal dichalcogenide (TMD) nano sheet is gaining popularity in the field of tribology. Calcination was used to create the graphitic carbon nitride (g-C_3N_4) nano sheet, and microwave assistance was used to create the nanocomposite with molybdenum disulfide (MoS_2).

I. Experimental

The chemicals used were brought from Aldrich and BDH and only doubly distilled water was used. The spectra were analyzed using the following instrumentation. A krypton ion laser from Spex Ramalog V with a Spex Datamate acquisition unit with KBr base at100 mW, along with a Perkin-Elmer 1720 Fourier-Transform spectrophotometer to generate IR spectra. Moreover, a Bruker WM-250 NMR spectrometer was used to elucidate NMR spectra and the Micro Analytical Department provided all microanalysis.

1.1. Preparation of manganese (II) complex:

In order to prepare the ligand complex, ethanol with 2.12g (0.01mol) of benzoin and the same molar amount of ethanoic solution of 1.98g (0.01mol) of Cr (II) and 1.25 g (0.01mol) Cu (II) salt were mixed together and brought to a rapid boil. Thereafter, the solution pH was maintained by NaOH and the mixture was allow to reflux for two hours before being washed and filtered to remove any material that was unreacted. The final product was assayed using TLC and confirmed with elemental analysis techniques. A product yield of 85% was obtained and a brown color complex was produced.

1.2. Preparation of copper (II) complex

An approximate 1:2 molar ratio respectively, of ethanolic solution of ligand at 15 milters (1.02g, 3.11mmol.) was added to 10 milters of ethanolic solution with cupper (II) chloride (1.09g, 6.39mmol) while heating under a stir for 2 hours. The final product was purified via recrystallization using ethanol after being washed and filtered.

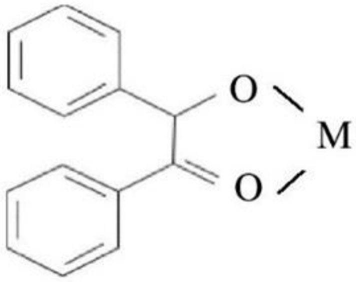
1.3. Physical measurements:

Carbon and hydrogen microanalyses were performed at PMU Saudi Arabia analytical center. A Conductivity meter model CMD650 digital was used to obtain molar conductance measurements. The percent electronic and infrared spectrum were gathered using Perkin-Elmer Lambda 4β and IFS-25 DPUS/IR (Bruker1988Y with KBr disk) Infrared Spectrophotometers, respectively.

Sample preparation for IR Analysis: In order to give the good results the homogeneous mixture of the sample was added to finely powdered potassium bromide.

1.4. Determination of Metal content:

Metal content determination was performed using a digestion technique where the solution of the complex was titrated with 0.01M EDTA, with Murexide indicator and ammonia buffer solution. Hence, the elucidation of the Cr (II) and Cu (II) content were determined to be 14.80% and 12.35%, respectively.



Structure of Benzoin Where M= Cr, Cu.

1.5. Antimicrobial Experiments

Antimicrobial activity. Both Gram-positive and Gram-negative bacterial strains were used to determine the antibacterial activity of the complexes formed by broth dilution method as per (CLSI) guidelines. Subsequently, such bacteria employed in this study included the following: *Bacillus cereus, Staphylococcus borealis, Xanthomonas* oryzae, *Rhodococcus* sp, *E.coli, Pseudomonas Fluorescens.* Pure bacterial stock was used with Sterile Mueller-Hinton broth media under 4°C conditions. Moreover, the solvent dimethyl sulfoxide were used as test compound dissolution followed by inoculation. The test sample compounds added were 0.25, 0.5, 0.75, 1.0, 1.25, 1.5, and 1.75 molar concentrations, respectively and measured against non-test sample compounds as controls. The sample solutions were incubated at 37° and the broth was measured at 600 nm after 24 hrs. Hence, from these experiments the decrease in the percentage absorbance was measured

II. Results and Discussion

Noted below are the results of the experimental micro elemental analysis (C.H.N). Also included in the results are the physical properties of compounds that were obtained in the experiment.

2.1. Vibrational spectra

In figures (1, 2 and 3) the ligand and the corresponding complex ion FT-IR spectra are displaced. The broad bands at (3415) cm^{-1} (3415) cm^{-1}, (3403) cm^{-1} and (3401) cm^{-1}, were recorded for υ (OH) of phenolic group, respectively [7]. Aromatic bands v(C-H) of chromium (II) and copper (II) complex were assigned at (3013) cm^{-1} and (3063) cm^{-1}. The vibration (stretching) of (-OCH$_3$) group for chromium (II) and copper (II) complex were identified in the spectra at (2833) cm^{-1} and (2841) cm^{-1} bands [8, 9]. Henceforth the spectra bands at (2717) cm^{-1} and (2737) cm^{-1} were respectively attributed to υ(C-H) vibrations of chromium (II) and copper (II) complex [10, 11 and 12]. The spectra at (1667) cm^{-1} was analysed for benzoin ligand corresponding to υ(C=O vibrations, and the shifts to the higher wavelengths of (1671) cm^{-1} and (1682) cm^{-1} correspond to Cr(II) and Cu(II) complexes suggested that there is ligand to metal coordination via the C=O group [13]. In benzoin complexes of Cr (II) and Cu(II) there were new band appearances in the regions (589) cm^{-1}, (485) cm^{-1} and (507) cm^{-1}. This emergence of the υ(M-O) was thought to be due to the formation of metal complexes by the coordination of the carbonyl groups [14]. The FT-IR spectral data is tabulated in Table (1) and the spectral vibrations of the ligands complexes.

2.2. Electronic Spectra Ligands and magnetic Susceptibility for the Complexes

The electronic spectral data of the ligand and its complexes with the metals are listed in Table (2). The ligand (C_6H_5COCH (OH) C_6H_5) at (3 M) in ethanol mentions one $\pi - \pi^*$ transition at (257 nm) and the three peaks at (307 nm), (337 nm) and (387 nm) correspond to ligand in the electronic spectra [9], and is shown in Figure (4). The ultra violet visible spectra of chromium and cooper are shown in Figures (5, 6). With both metals $\pi - \pi$ and n– π^* transitions shifts and band intensity changes were observed in the complexes in response to ligands. Thus, this observed change shows the metal-ligand

Table 1.

Analytical and spectroscopic data of Benzoin, Chromium and Copper Benzoin complexes

Compound	Found (Calcd) %			I.R (cm^{-1})					
	C	H	O	υ(C=O)	υ(HCO)$^+$	υ(C=C)	υ(C-H)	υ^s(MO$_2$)	υ^{as}(MO$_2$)
[PhCH(OH)C(O)Ph]				1589s (1570s)^y	1501m,	1341s	1199s (1201s)^c		
[PhC(O)C(O)PhCrPhC(O) C(O)Ph]H$_2$O	20.1 (21.7)	10.1 (12.5)	6.0 (8.1)	1570m	1489s,	1327 m	1231s	901s	961s
[PhC(O)C(O)PhCuPhC(O) C(O)Ph]2H$_2$O	32.7 (34.5)	11.1 (12.9)	7.0 (8.5)	1569s	1479s,	1315s	1195s	902s	851s

^{a}Na, K analysis, and ^{y}Raman data.

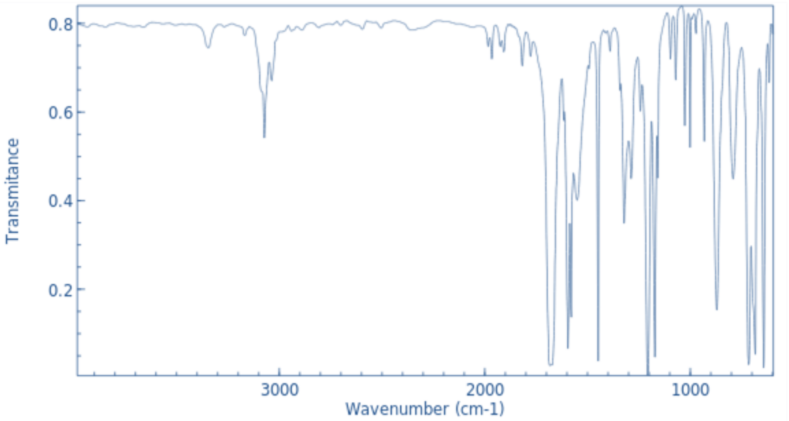


Fig. 1. FT-IR spectrum of the Benzoin

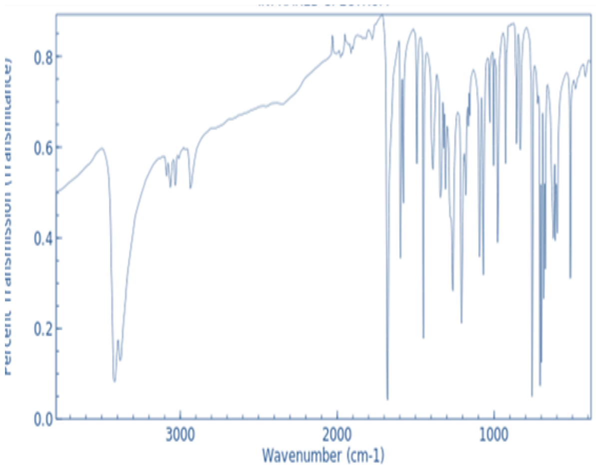


Fig. 2. FT-IR spectrum of the Chromium Benzoin

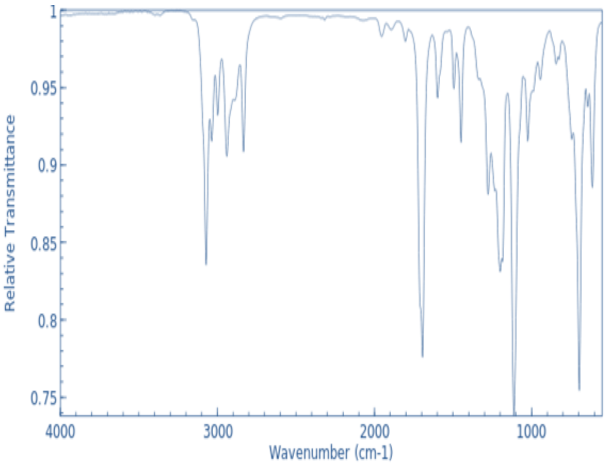


Fig. 3. FT-IR spectrum of the Copper Benzoin

Table 2.

UV-Reflectance and Magnetic data

Compounds	**Absorption λmax (nm)**	**Assignment**	**Magnetic Moment (μeff (B.M.)**	**Possible Geometry**
[PhCH(OH)C(O)Ph]	307	n→π*		
[PhC(O)C(O)PhCrPhC(O)C(O)Ph]H$_2$O	337	^{4}A2→^{4}T1(P)	1.56BM	Square-planar
[PhC(O)C(O)PhCuPhC(O)C(O)Ph]^{2}H$_2$O	387	^{2}B1(g)→ ^{2}A1(g)	1.64BM	Square-planar

coordination. The high intensity peaks for (Cr) complexes at (358 nm) is mostly because of charge transfer transition [15]. The visible range contains peaks that are related to d-d transitions. In the 788 nm wave length for the Cu-complex suggests square planar geometry for Cu (II) ion with the assigned to $^2B_1g \rightarrow {}^2A_1g$ [16]. Moreover, further square-planar geometry assignments were made for complexes (Cr and Cu) for the magnetic moment values of 1.56 BM and 1.64 BM, respectively [17].

2.3. ^{1}H and ^{13}C NMR spectra of Benzoin, Chromium and Copper Benzoin complexes

The Proton NMR spectra of $C_6H_5COCH(OH)C_6H_5$ in Table 3 predicts the splitting of H_3 and H_6 protons with

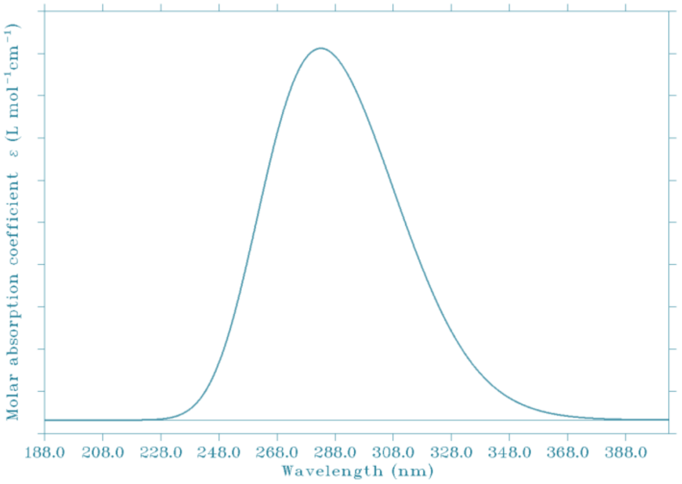


Fig. 4. UV spectrum of the Benzoin.

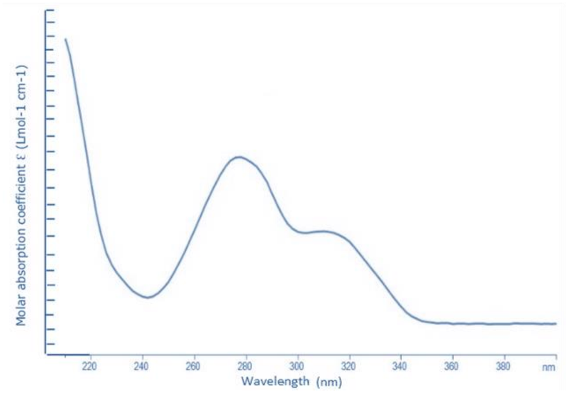


Fig. 5. UV spectrum of the Chromium Benzoin

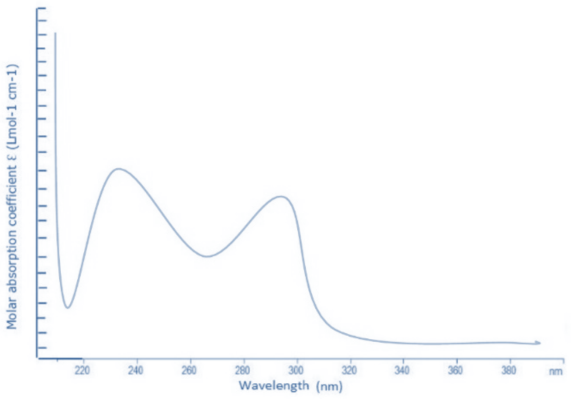


Fig. 6. UV spectrum of the Copper Benzoin.

Table 3.

^{1}H-NMR Spectral data of Benzoin, Chromium and Copper Benzoin complexes.

Benzoin		Chromium Benzoin		Copper Benzoin	
(δ) ppm	**Assignment**	**(δ) ppm**	**Assignment**	**(δ) ppm**	**Assignment**
(3.51)	(-OCH$_3$)	(3.1)	(-OCH$_3$)	(3.67)	(-OCH$_3$)
(6.01)	(-OH)	(6.1)	(-OH)	(6.7)	(-OH)
(6.81 - 7.01)	(Ar-H)	(7.15 - 7.27)	(Ar-H)	(7.11 - 7.37)	Ar-H)

Table 4.

^{13}C NMR Spectral data of Spectral data of Benzoin, Chromium and Copper Benzoin complexes.

Compound	$C_{3,6}$	$C_{1,2}$	$C_{4,5}$	Solvent
[PhCH(OH)C(O)Ph]	101.0	167.0		$(CD_3)_2SO$
PhC(O)C(O)PhCrPhC(O)C(O)Ph]	99.5	177.3	179.3	D_2O
[PhC(O)C(O)PhCuPhC(O)C(O)Ph]2H$_2$O	98.2	171.1	175.5	CD_3Cl

singlets having values of δ 6.7 ppm and the hydroxyl protons was identified as a broad singlet with δ 6.01 ppm value. Although the protons of the complexes with hydroxyl group cannot be observed as there is signaling due to the resonance from H_3 and H_6 protons being shifted left towards smaller values [18]. This shift is thought to be due to the decline in the electron density of the coordination ring. The ^{1}H-NMR spectrum of Benzoin, Chromium and Copper Benzoin complexes are shown in Figure 7, 8 and 9.

The chromium-benzoin complex ^{13}C NMR spectrum (Table 4) shows a sharp resonance at δ104.0 ppm followed by a broad resonance at δ169ppm. It is speculated that these resonances arise due to $C_{3,6}$ and $C_{1,2,4,5}$ (carbon and oxygen atoms) respectively. The complex copper-benzoin in Table 4 show ^{13}C NMR at 3 resonances near δ102.0, 177.0 and 181.0 ppm, thought to be from $C_{3,6}$, $C_{1,2}$ and $C_{4,5}$ [19]. The data shows that there are at least 3 carbon types among the complexes. Moreover, carbon atoms near to oxygen atoms ($C_{1,2}$ and $C_{4,5}$) show resonances that are shifted downfield by coordination.

2.4. Antimicrobial activity studies

The ligands in free-state and in combination with the metals was done against microbial strains viz, *Bacillus cereus*, *Staphylococcus borealis*, *Xanthomonas* oryzae, *Rhodococcus* sp, *E.coli*, *Pseudomonas Fluorescens*. As per the results the ligand in free-state shows low antimicrobial activity, which could be ascertained due to unattached hydroxyl groups and other substituents which could inhibit the development of microorganisms by making a change in the growth and metabolism of the bacterial cell wall and the bacterial cell as whole. However, the complexes of the concerned metals with benzoin show good response as antimicrobial agents against the mentioned microorganisms may be because of the charge transfer that occurs between the metal and ligand. The results demonstrated that with an increase in the concentration of the complex ions, in which maximum inhibition was observed there was also an increase in the antimicrobial activity. As per the results, it was noted that an increase with concentration shows increase in antimicrobial activity although maximum inhibition was observed for the *Rhodococcus* sp, followed by *Bacillus cereus*, and *E. coli*. Among the complex ions it had been found that [PhC(O)C(O)PhCuPhC(O)C(O)Ph]$2H_2O$ shows more inhibition as compared to [PhC(O)C(O)PhCrPhC(O)C(O)Ph] and the sole ligand

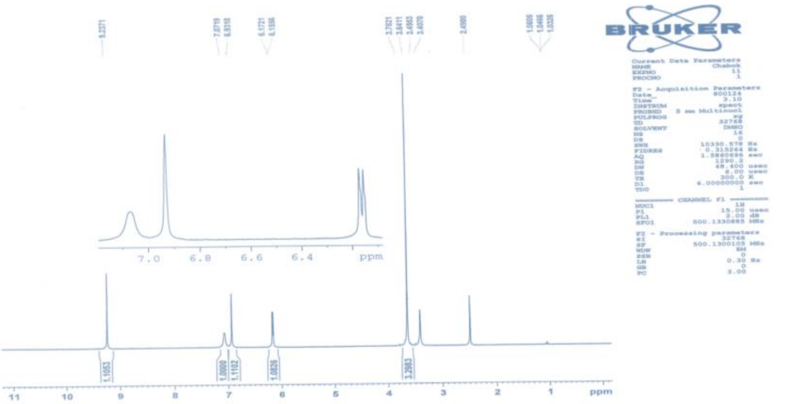


Fig. 7. ^{1}H-NMR spectrum of Benzoin.

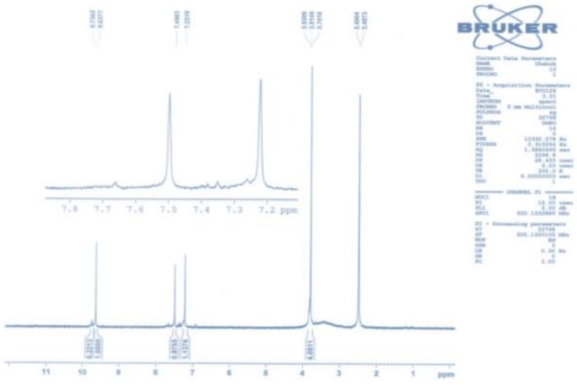


Fig.8. ^{1}H-NMR spectrum of the Chromium Benzoin.

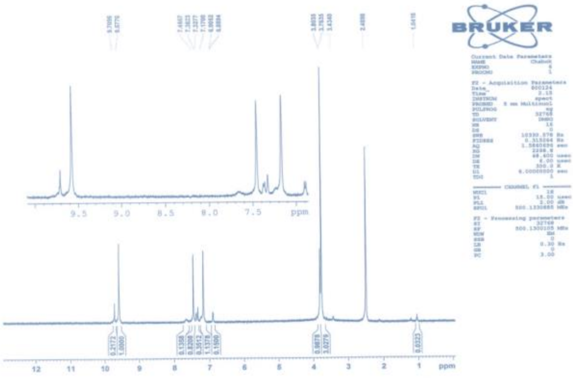


Fig. 9. ^{1}H-NMR spectrum of the Copper Benzoin..

Table 5.

Antimicrobial activity data of the Benzoin, Chromium Benzoin, Copper Benzoin, Azithromycin

Test Organisms	**(%) of Complexes**												
	Benzoin				Chromium Benzoin				Copper Benzoin				Azithromycin
	25%	50%	75%	100%	25%	50%	75%	100%	25%	50%	75%	100%	50%
Bacillus cereus	1 (0.3)	1.8 (0.9)	2.4 (0.8)	3.5 (0.8)	0.9 (0.5)	1.4 (0.2)	1.9 (0.9)	2.8 (0.9)	1.4 (0.8)	1.9 (0.5)	2.5 (0.7)	3.5 (1)	**5.2**
Staphylococcus borealis	0.6 (0.19)	1.3 (0.4)	1.7 (0.9)	2.5 (0.9)	0.5 (0.2)	1 (0.4)	1.5 (0.7)	2.6 (0.9)	0.8 (0.3)	1.5 (0.5)	1.9 (0.6)	2.9 (1)	4.5
Xanthomonas oryzae	0.05 (0.02)	0.07 (0.02)	0.5 (0.3)	0.8 (0.7)	0.2 (0.1)	0.4 (0.2)	0.7 (0.4)	1 (0.5)	0.07 (0.03)	0.2 (0.1)	0.5 (0.1)	1 (0.4)	3.4
Rhodococcus sp	0.4 (0.2)	1.5 (1.1)	2.2 (1.9)	2.9 (2.3)	0.8 (0.5)	1.3 (0.5)	1.3 (1)	3.2 (2.3)	1.5 (1.2)	2.8 (2.1)	3.7 (2.1)	3.9 (3.1)	5.2
E.coli	0.9 (0.4)	1 (0.7)	1.8 (1.3)	2 (1.9)	0.8 (0.4)	1.5 (0.5)	2.2 (1.3)	3.3 (2.1)	2.5 (1.9)	2.5 (2.1)	3 (2.3)	3.5 (2.5)	5.4
Pseudomonas fluorescens	1.5 (0.8)	1.7 (0.9)	0.5 (0.3)	1.5 (1.1)	0.4 (0.2)	1.5 (0.5)	2.7 (2.1)	3 (2.1)	1.5 (1.1)	1.9 (1.3)	2.5 (2.0)	3.3 (2.1)	4.5

∗ *Ligands data between parentheses.*

against the mentioned microbes as shown in table 5.

Conclusion

In conclusion, the coordination complexes of Cr and Cu with Benzoin were synthesized in high yield. The complexes were then analyzed and characterized by various spectroscopic techniques that included the following: viz, infrared, Ultraviolet, ^{1}HNMR, ^{13}CNMR. The complexes clearly indicated that formation takes place by 1:1 ratio between the metal and the ligand. The complex ions and the ligand were also magnified for determination of antimicrobial activity, where the complex species showed good response against the concerned microbes. Hence, the results show that the complexes perform well in suppressing the growth of the microbes.

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Синтез та спектральний аналіз комплексів хрому та міді 2-гідрокси-1, 2-дифенилетан-1

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Для аналізу комплексів хрому (II) і міді (II) з бензоїном використовуються різні хімічні процеси. Вищезазначені комплекси проаналізовано щодо елементного аналізу та охарактеризовано ІЧ та електронними спектрами, ЯМР спектроскопією. Аналіз даних показав утворення іонів металів у співвідношенні 1:1 ліганд до металу, що є іонним комплексом [Cr (B)] H_2O та [Cu (B)] $2H_2O$, у якому B є бензоїном. Згідно з даними, проаналізованими за допомогою інфрачервоної спектроскопії, саме через карбонільний і гідроксильний кисень бензоїну відбувається хелатна хімічна реакція. Комплексний структурний аналіз досліджено за допомогою електронного спектрального аналізу даних і протонної ЯМР-спектроскопії. Досліджувані складні системи із металами демонструють також антимікробні властивості щодо різних штамів бактерій.

Ключові слова: Бензоїн, Координаційний комплекс, Хром, Мідь, Антимікробні властивості.