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Synthesis, Characterization and Antimicrobial Activity of Pd(II) and Cu(II) Complexes of Schiff base Derived from Phenylethyl amine

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Abstract

Condensation of phenyl ethyl amine with 2-Hydroxy-3-naphthaldehyde yielded Schiff base derivative in good yield. Palladium and copper were selected for preparation of complexes. The structures of new compounds were assigned by FTIR and ^{1}H NMR spectroscopy. The synthesized compounds were tested for their antibacterial activity against Staphylococcus aureus, Escherichia coli, Bacillus cereus, and Klebsella pneumonia. Additionally, they evaluated for their antifungal activity against Candada albicans, Candada trobicalis, candida krusi, Aspergillus multi, and Aspergillus niger. All compounds revealed significant antibacterial and antifungal activity.

Key words: Phenylethyl amine, 2-Hydroxy-3-naphthaldehyde, Schiff base, Microbial activity, HSQC-NMR

Introduction

Phenylethylamine or β-phenethylamine is a trace amine, an organic compound, and a natural mono amine alkaloid. Phenylethyl amine well known for psychoactive drug and stimulant effects(1). It functions as a neuromodulator or neurotransmitter in the mammalian central nervous system(2). The carbon-nitrogen double bond chemistry plays a vital role in progresses of chemistry science. Schiff base reveals activity against tubercular, cancer, bacterial, fungal, analgesic, CNS depressant, inflammatory, convulsant, insecticidal, mouse hepatitis virus (MHV), herpes simplex virus type 1 (HSV-1) and adenovirus type 5 (Ad 5), mosquito larvae and herbicidal activities(3,4). Previously, the biological activity for Pd(II) complexes containing S and N donor ligands on several tumor lines was investigated(5). This work aimed to synthesis new complexes Schiff base derived from phenylethyl amine and 2-Hydroxy naphthaldehyde (Scheme l) and study their antimicrobial activity.

Material and Method

Physical measurements

The IR spectra were recorded on a Pye-Unicam SP3-300 spectrometer using KBr discs at Department of Chemistry, College of Education for Pure Sciences, University of Basrah, Iraq. 1H, 13C and 2D NMR spectra were measured on a Bruker at 600 MHz with TMS as internal reference at Konstanz University, Germany. Melting points were measured by a Philip Harris melting point apparatus at College of Veterinary medicine, University of Basrah, Iraq.

Synthesis

Synthesis of Schiff base[6] 1

Phenyl ethyl amine (0.4 g, 13.36 mmol) in ethanol(25ml) was added to (0.58g, 13.36 mmol) of hot ethanolic solution of 2-hydroxy naphthaldehyde. Then two drops of glacial acetic acid was added. Resulting solution was heated under reflux for 3h and stand overnight in refrigerator. After that the solid product obtained was filtered, washed with acetone, and recrystallized by using chloroform: ethanol (8:2 ratio) to yield yellow crystal of (E)-1-[(2-phenylethyl) carbonoimidoyl] naphthalene 2-ol.

Yield:80%,M.p.:192-194°C.

FT-IR (KBr, ν, cm^{-1}): 3300(OH); 3068,3024(CH-aromatic); 2929, 2858(CH- aliphatic); 1639-1537(C=C, C=N).

^{1}HNMR(600MHz,$CDCl_3$,δ,ppm):14.35(s,1H,OH); 8.39 (s, 1H, CH=N);6.80(d.1H,$H_{arom.}$ 3^-); 7.15-7.19(m,4H, $H_{arom.}$ 3,5,6^-,9);7.26(t,1H, $H_{arom.}$ 4);

7.29(t, 2H, $H_{arom.}$ 7^-,8^-);7.48(d.1H,$H_{arom.}$ 4^-);5.57(d.1H,$H_{arom.}$ 5^-);6.82(d.2H,$H_{arom.}$ 2,6)

3.73 (t, 2H, CH_2-N), 2.93 (t, 2H, CH_2-Ar).

^{13}C NMR (600 MHz, $CDCl_3$, δ, ppm), 37.40 (CH_2-Ar), 54.72 (CH_2-N), 106.48-137.96

(C-Ar), 158.01 (C-CH=N), 176.47 (C-OH).

Synthesis of copper complex 2

Schiff base (5mmol) was dissolved in 20ml of ethanol solution. Copper(II)chloride (2.5mmol) was then added. A stirrer was then inserted and the reaction mixture heated to 75-80^{0}C for 2h. After that, the precipitate was filtered, washed with ethanol, and dried in oven at 70^{0}C to yield green crystals of copper complex. Yield; 73%, M.P.= 202-205 ^{0}C. FT-IR (KBr, ν, cm^{-1}): 3400(OH); 3068,3022(CH-aromatic); 2927, 2862(CH-aliphatic); 1631-1530(C=C, C=N). ^{1}H NMR (600 MHz, DMSO 6d, δ, ppm):14.05(s,2H,OH); 9.03 (s, 2H, CH=N);6.71-7.72(m, 22H, Ar-H); 3.92 (t, 2H, CH_2-N), 2.99 (t, 2H, CH_2-Ar). ^{13}C NMR (600 MHz, DMSO 6d, δ, ppm), 36.96 (CH_2-

Ar), 52.64 (CH_2-N), 106.14-138.95 (C-Ar), 159.50 (C-CH=N), 177.77 (C-OH).

Synthesis of palladium complex 3

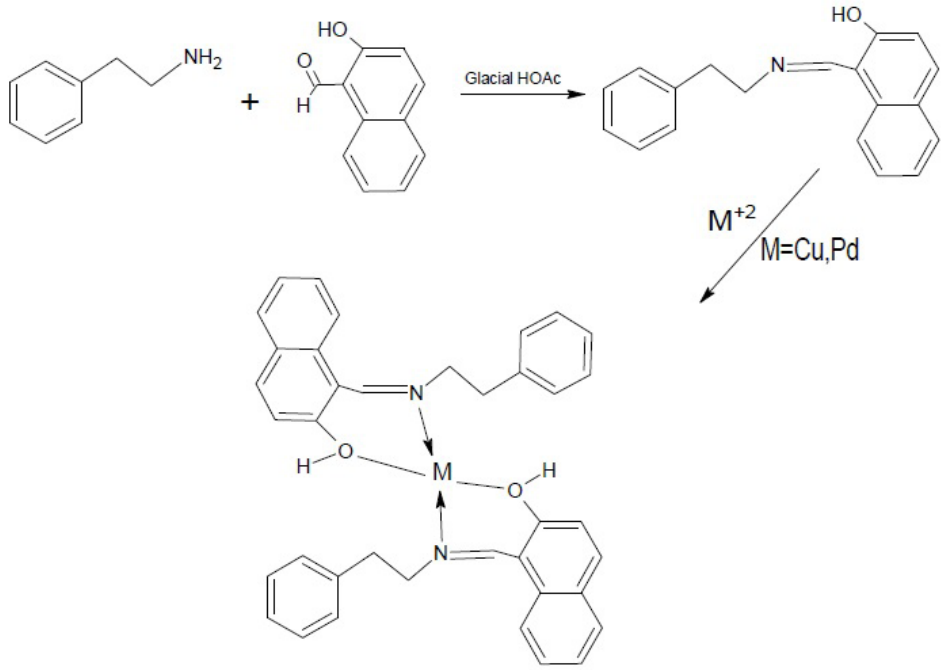
Palladium(II) complex was prepared using palladium(II)chloride instead of copper(II)chloride to yield yellow-brown crystals of palladium complex. Yield; 77%,

M.P.= 225-227 ^{0}C. FT-IR (KBr, ν, cm^{-1}): 3350(OH); 3065,3027(CH-aromatic); 2931,

2859(CH-aliphatic); 1637-1539(C=C, C=N). ^{1}H NMR (600 MHz, DMSO 6d, δ, ppm):12.00(s,2H,OH); 10.83 (s, 2H, CH=N);7.04-7.99(m, 22H, Ar-H); 3.17 (t, 2H,

CH_2-N), 2.07 (t, 2H, CH_2-Ar). ^{13}C NMR (600 MHz, DMSO 6d, δ, ppm), 36.58 (CH_2-Ar), 56.58 (CH_2-N), 110.54-141.76 (C-Ar), 160.10 (C-CH=N), 171.07 (C-OH).

Scheme 1: Preparation of palladium and cupper complexes derived from phenylethyl amine.



Antimicrobial activity

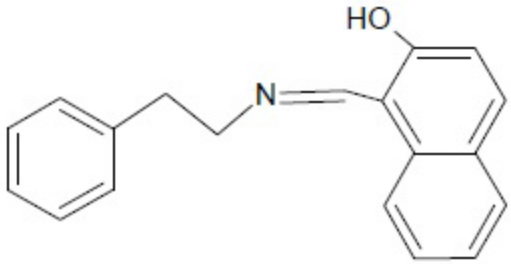
In vitro, the synthesized compounds (1-3) were tested for their antibacterial activity against Staphylococcus aureus, Escherichia coli, Streptococcus, Bacillus cerius, salmonella, klebsella, and Pseudomonas b y using disc-agar diffusion method[7]. For antibacterial activity, Muller Hinton Agar (20 ml) was used as culture media in petri dish. The synthesized compounds (1-3) were also tested for their activity against Candida albicans, Candida trobicalis, Candida krusi, Apergillus multi and Aspergillus niger by using the same method. For antifungal activity, Sabouraud's dextrose agar (20 ml) was used as culture media in petri dish. Concentrations at 100, 200 and 300 µg/ml of the test samples in DMSO solvent was introduced in the respective method. Filter paper (sterile Whatman No. I) disks (6mm in diameter) was soaked with the solution in dimethylsulfoxide (DMSO) and put on the Petri plates. I n negative control, a paper disc was soaked with (DMSO). The plates were then incubated a t 3 7 °C for 24 h and at 28°C for 72 h for bacteria and fungi, respectively. The inhibition zone diameters were measured in millimeters by using a caliper **vernia.**

Findings

In current study, Cu(II) and Pd(II) complexes of• Schiff bases was synthesized. To produce the Schiff base derivative (L) Scheme 1 in good yield, the reaction of ethyl phenyl amine with 2-hydroxy naphthaldehyde (1:1 ratio) was used. The ligand (L) reacted with metal ions(II), Cu, and Pd forming complexes was done (2:1 ratio). The FT IR and NMR spectral analysis were used to confirm their structures. The presence of the azomethine group (-CH=N) stretching with a sharp region around revealed by the FT IR spectra 1530-1539 cm-1 due to azomethine protons (CH=N) at 8.39, 9.03 and 10.83 ppm respectively, all compounds (1-3) showed signal by using1 H NMR spectra. The high field of complexes due to the high electronegativity of metals of all compounds showed a triplet at the range 2.07-2.99 ppm due to CH2-Ar and triplet at range 3.17-3.92 ppm due to CH2-N group. The region at 6.80- 7.99 ppm was due to aromatic protons. The synthesized compounds (1-3) showed singlet's at 14.35, 14.05, and 12.00 ppm, respectively due to phenolic groups Ar-OH. All compounds were measured in DMS0-d6 by using 1HNMR spectrum. In addition, the formation of these compounds was provided by 1HNMR spectra. The presence of CH=N group around 158.01-160.0 ppm was revealed by the spectra. The signals around 171.07-177.77 ppm was due to C-OH. These spectra data sports the structure of synthesized compounds. The 1H, 13C HSQC-NMR spectrum(8) of compound 1 showed a cross peak at δH/δC =14.35/178.4 ppm, belonged to Ar-OH. The cross peak at δH/δC =8.39/158 ppm due to azomethine group (N=CH), Thus, the correlation of protons and carbon in aromatic rings such as δH/δC = 7.57 /137, 7.48/129 ppm and other positions can be assigned to the protons and carbon atoms of the aromatic rings(9) (Table 1).While, the cross peak at δH/δC= 3.73/54.7 and δH/δC =2.93/37.4 ppm might be attributed to methylene groups(Table l, Figure 1). The 1H, 13C HSQC- NMR spectrum of copper complex 2 showed a cross peak at δH/δC = 9.03/159.5 ppm due to azomethine group (N=CH). Thus, the correlation of protons and carbon in aromatic rings such as δH/δC= 8.00 /118, 7.71/137.48 ppm and other positions can be assigned to the protons and carbon atoms of the aromatic rings (Table 2). While, the cross peak at δH/δC =3.90/52.64 and δH/δC =2.99/36. 96 ppm can be attributed to methylene groups, (Table 2, Figure 2).

Table1 : H, C -HSQC NMR data of 1-[

(2-phenylethyl) carbonoimidoyl] naphthalene2-ol.

Compound Structure	δH(ppm)	δC(ppm)	Position
	14.35	178.4	C-OH
	8.39	158	C,H(CH=N)
	7.57	137	C,H(3)
	7.48	**129**	C,H(4-)
	7.29	128.9	C,H(7-,8-)
	7.13	128	C,H(6-,9-)
	7.26	128.1	C,H(4)
	7.15	127.9	C,H(3,5)
	6.82	106.4	C,H(2,6)
	3.73	54.7	C,H(CH_2-N)
	2.93	37.4	C,H(CH_2-Ar)

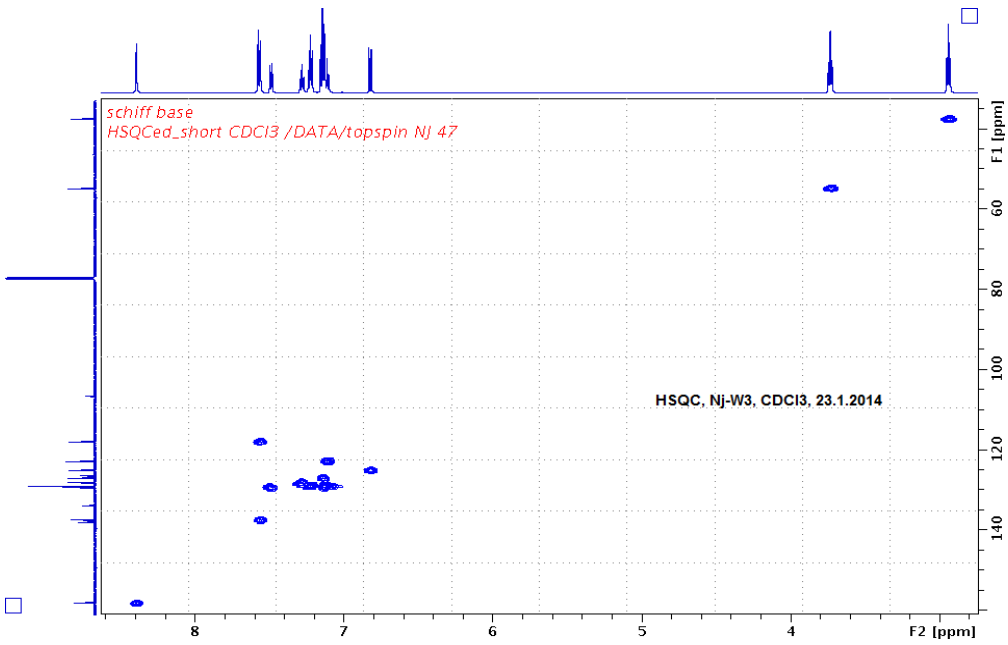


Fig 1: $^{1}H,^{13}C$-HSQC NMR data of 1-[(2-phenylethyl)carbonoimidoyl]naphthalene2-ol.

Table 2: 1H, 13 C- HSQC NMR data of copper complex

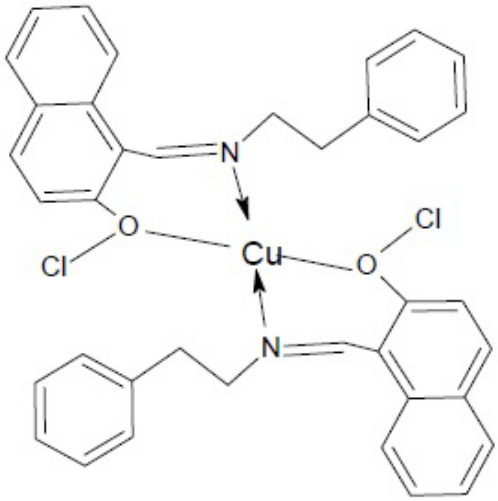
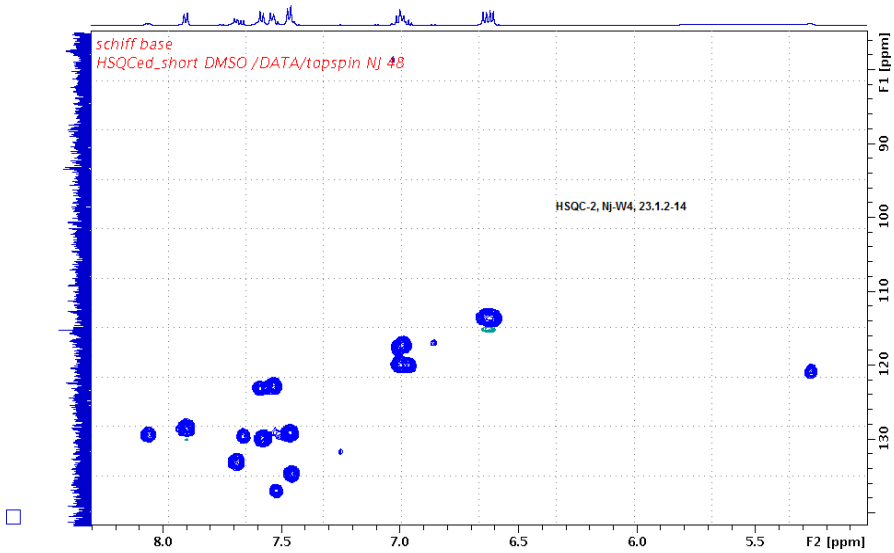
Compound Structure	δH(ppm)	δC(ppm)	Position
	9.03	159.5	C,H(CH=N)
	8.00	118.87	C,H(3-)
	7.71	137.48	C,H(4-)
	7.29	129.34	C,H(6-,9-)
	7.43	128	C,H(7-,8-)
	7.35	129.36	C,H(4)
	7.18	129.32	C,H(3,5)
	7.14	122.59	C,H(2,6)
	3.90	52.64	C,H(CH2-N)
	2.99	36.96	C,H(CH2-Ar)

Fig 2: ^{1}H, ^{13}C-HSQC NMR data of copper complex

The activity of the synthesized compounds against bacteria a n d fungi was evaluated by using the agar disk



diffusion method [7]. The current study revealed that the compounds activities elevate against bacteria and fungi

with an increase the solution concentration (Table 4 and Table 5). The synthesized compounds showed low activity against *E. coli,* S.*aureus,* and *Pseudomonas* for Schiff base (L) and copper complex, but palladium complex showed good antibacterial activity against *salmonella, klebsella* and *Pseudomonas*(Table 4).

Table 4: Antibacterial activityofthe Schiff-baseand it complexes

Comp.	Conc. µg/ml	S.aureus			*E.Coli*			Streptococcus			*B.Cerus*			Salmonella			pseudomonas			Klebsella		
		100	200	300	100	200	300	100	200	300	100	200	300	100	200	300	100	200	300	100	200	300
Ligand		-	-	7	-	-	7	-	-	-	-	-	7	-	-	-	-	-	7	-	-	-
Co(II) complex		-	-	7	-	-	-	-	-	-	-	-	-	-	-	-	-	-	8	-	-	-
Pd(II) complex		-	-	-	-	-	-	-	-	-	-	-	-	-	-	15	-	7	9	-	-	10

***Results are expressed the diameter of inhibition zone in mm for different bacterial species**

Table 5: Antifungal activity ofthe Schiff-base and it complexes

Comp.	Conc. µg/ml	C.albicans			C.trobicals			C.krusi			A.multi			A.niger		
		100	200	300	100	200	300	100	200	300	100	200	300	100	200	300
Ligand		11	15	15	10	12	15	10	13	15	-	-	-	-	-	-
Co(II) complex		-	-	-	-	-	-	-	-	-	-	-	7	-	7	7
Pd(II) complex		-	-	11	-	-	12	-	-	10	-	-	10	-	7	10

***Results are expressed the diameter of inhibition zone in mm for different bacterial species**

Conclusion

A Schiff bases of phenyl ethyl amine and their copper and palladium complexes were characterized by using Infra red (FTIR) and nuclear magnetic resonance (NMR) spectroscopy. The antimicrobial activity and antifungal activities were evaluated against bacterial (seven strains) and fungal (five species), respectively. The Schiff base compound revealed better antifungal activity compare with metal complexes. These results are promoted us to continue the study such as, physiological parameters and histopathological study to complete the pharmaceutical studies.

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Conflict of Interests: The authors declare that they have no conflict of interest.

Source of Funding : No funding (Authors self)

Ethical Clearance: All ethical guidelines have been adhered according to committee on the ethics of dealing with laboratory animals.

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