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New 3- hydrazonoindolin-2-one Cd(II) complexes with amino pyridine ligands, Synthesis, Characterization and biological activity evaluation

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ABSTRACT

his research includes synthesis and characterization of some of Cd(II) complexes with (3-hydrazonoindolin-2-one)(HZI) ligand and amino pyridine ligands.

Treatment equialmolar of $CdCl_2.2.5H_2O$ and (HZI) ligand with two moles of n-aminopyridine (n-amp) (n:2,3,4) ligands afford a tetrahedral complexes of the type $[Cd(HZI)(n-amp)_2]Cl_2$, where (HZI) ligand behaves as a bidentate chelating fashion through the N atom of azomethine group and O atom of carbonyl group. Whereas the (n-amp)(n: 2,3,4) was bonded monodentate mode through the N atom of heterocyclic ring.

The prepared complexes have been characterized by molar conductivity, elemental analysis, infrared spectra and ¹H-NMR and ¹³C-NMR spectra. Also the evaluation of biological activity of the prepared complexes against two types of gram positive bacteria (*Staphylococcus Epidermidis* and *Staphylococcus aureus*) and (*Citrobacer Freundii*) and gram negative, all prepared complexes showed activity against *Staphylococcus aureus* more than amikacin, while the [Cd(HZI)(3-amp)₂]Cl₂ complex showed high activity against *Staphylococcus Epidermidis* and *Citrobacter freundii* more than another prepared complexes.

Introduction

Isatin or Tribulin is a derivative of indole from heterocyclic compounds, and its systemic name (1H-indole-2,3-dione), which was obtained by the scientists Erdman and Laurent in 1840 [1] as a product of the oxidation of Indigo (It is a distinctive blue dye) by nitric acid and chromic acid [2]. The isatin is present inside the human body in the brain and many body tissues and fluids. It also has many biological activities such as causing activities, Antispasmodics, analgesics, anti-convulsions and as a potent against of the receptors atrial peptide in vitro [3,4], antibacterial [5,6], anti-HIV activities [7,8], as well as liver metabolites, are found in nature in plants such as mushrooms [9].

An Isatin use as ligand alone in preparation of many complexes or it was prepared a new isatin derivatives such as shiff base, Hydrazine-derived isatins were found to be active against sarcoma, antibacterial and antifungal [10,11]. Similarly, acetone- and ketone-derived isatins exhibited anticonvulsant activity [12].

Therefore in this study, a new Cd(II) with (3-hydrazonoindolin-2-one) (HZI) with n-aminopyridine as co-ligand was synthesized and characterized and studied the biological properties.

Experimental

Materials and methods

All chemical materials and solvents were supplied and used without purification. C.H.N analysis was recorded on an Elementar vario El III C.H.N elemental analyzer. The Nuclear magnetic resonance was measured on a Bruker 400 MHz spectrometer in DMSO-d⁶ as a solvent. The melting point of the ligands and prepared complexes was recorded on Automatic (SMP30) melting point apparatus. The infrared spectra of compounds were recorded with Shimadzu FT-IR 8400S KBr using spectrophotometer in the 400-4000 cm⁻¹ range. The molar conductivity of 10⁻³ M Freshly DMSO solution of prepared complexes was measured by using (Starter 3100c) digital conductivity meter.

Preparation of 3-hydrazonoindolin-2-one (HZI)



A solution of 98% hydrazine (0.055g, 1.1mmol) in (10ml) of absolute ethanol was added to a solution of isatin (0.161g, 1mmol) in (10ml) of absolute ethanol ethanol with a few drops from glacial acetic acid. The mixture was refluxed for an hour, and then cooled to room temperature, where a yellow precipitate was separated. The yellow ppt. produced was filtered and washed with cold ethanol and dried under vacuum and recrystallized from a mixture of EtOH / DMF [13].

Preparation of the [Cd(2-amp)₂Cl₂] complex

A solution of $CdCl_2.2.5H_2O$ (0.342g, 0.00015mol) in EtOH (10 ml) was added to a suspension of 2-aminopyridine (2-amp) (0.282g, 0.003mol) in (5ml), The mixture was stirred at room temperature for an hour, then a white ppt. was formed. The white

product was filtered off, and dried under vacuum in oven (0.700g; 75.5%).

The $[Cd(3-amp)_2Cl_2]$ and $[Cd(4-amp)_2Cl_2]$ complexes were prepared and isolated in similar method.

Synthesis of [Cd(HZI)(2-amp)₂] complex

This complex was prepared by two different methods: **First**:

A solution of the HZI ligand (0.080g, 0.005mol) in EtOH (10ml) was added to a suspension of [Cd(2-amp)Cl₂] (0.138g, 0.005mol) in EtOH (10ml), The mixture was stirred for 2 hours at a room temperature. The light yellow precipitate was formed, then filtered and dried in the oven under vacuum (0.5130g, 66.7%).

The following complexes $[Cd(HZI)(3-amp)_2]Cl_2$ and $[Cd(HZI)(4-amp)_2]Cl_2$ were prepared and isolated in the similar method.

$$\begin{array}{c|c} & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & &$$

Scheme 1: Preparation of complexes

Second 2nd method:

A solution of $CdCl_2.2.5H_2O$ (0.342g,0.0015mol) (10ml) was added to a solution of 2-aminopyridine (2-amp) (0.282g,0.003mol) in EtOH (5ml), the mixture was stirred for an hour at room temperature where the white suspension was formed, then a solution of (HZI) (0.0015mol, 0.241g) in EtOH (10) ml of was added to the white suspension, the mixture was stirred gradually for 2 hours at room temperature where the yellow precipitate was formed, filtered and dried in the oven under vacuum (0.6152 g,77.8%).

The following complexes [Cd(HZI)(3-amp)₂]Cl₂ and [Cd(HZI)(4-amp)₂]Cl₂ were prepared and isolated in similar method.

Results and discussion

Molar electrical conductivity

The results of the molar electrical conductivity of the (10⁻³M) freshly solution of the (25°C) showed that. All the prepared complexes are electrical were in a molar ratio of (1: 2) positive ion: negative ion.

Table 1: Color, Yield (%), M.P., molar conductivity and Elemental Analysis of the Prepared ligand(HZI) and [Cd(HZI)(n-amp)₂]Cl₂ n=1,2,3 Compounds

Seq	Compound	Color	M.P.	W.t	Yield %	Λ	Found(cal)%		%
			C°	G		(ohm ⁻¹ .cm ² .mol ⁻¹)	C	H	N
1	HZI	Yellow	240-242	2	92	-	59.62	4.38	26.09
							(59.35)	(4.19)	(26.00)
2	Cl ₂]Cd(HZI)(2-amp) ₂ [Yellowish white	236-239	0.19	71	70.01	40.58	3.60	13.41
							(40.32)	(3.47)	(13.33)
3	Cl_2]Cd(HZI)(3-amp) ₂ [Yellowish white	233-235	0.20	75	73.76	-	-	-
4	Cl ₂]Cd(HZI)(4-amp) ₂ [Yellowish white	243-246	0.17	79	80.74	40.58	3.60	13.41
							(40.41)	(3.45)	(13.35)

Infrared spectra

The infrared spectrum of (HZI) ligand showed a new stretch band of the azomethine group $\upsilon(C=N)$ at (1589cm⁻¹) after the disappearance of the stretch band of $\upsilon(C=O)$ carbonyl group in the free isatin. The $\upsilon(C=O)$ carbonyl amide group showed at (1685cm⁻¹), while the $\upsilon(NH)$ group showed at (3153cm⁻¹) and the

symmetrical and asymmetric stretching of the $\nu(NH_2)$ group displayed at (3193,3353cm⁻¹) respectively [13]. The spectra of the prepared complexes showed the $\nu(C=O)$ carbonyl amide group bands within the (1685-1683cm⁻¹) range, the azomethine $\nu(C=N)$ group bands displayed within the (1606-1587cm⁻¹) range, while the $\nu(NH)$ group bands showed at 3157cm⁻¹), either symmetric and asymmetric $\nu(NH_2)$



bands displayed within the $(3207-3197\text{cm}^{-1})$ and $(3362-3353\text{cm}^{-1})$ range respectively. The $\upsilon(C=N)$

group in aminopyridine ligands were displayed within the (1552-1550cm⁻¹) range [14,15].

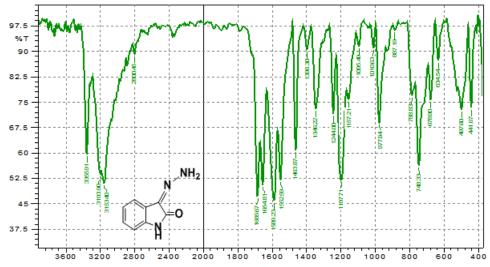


Fig. 1: IR spectrum of (HZI) ligand

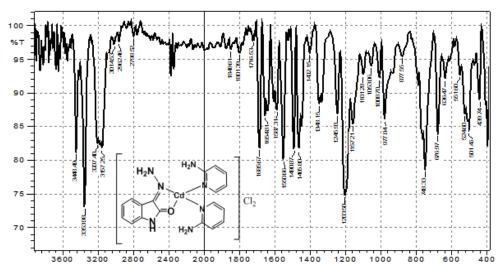


Fig. 2: IR spectrum of [Cd(HZI)(2-amp)₂]Cl₂ complex

Table 2: Selected IR stretching vibration bands (cm⁻¹) of the ligand and ids complexes

Compounds	HZI				Amine ligand			
	vC=O	vC=N	vNH	vNH_2	v _{Ar} C-H	vN=C	vNH ₂	
HZI	1685s	1589s	3153s	3193s	-	-	-	
				3355s				
Cl ₂]Cd(HZI)(2-amp) ₂ [1685s	1587s	3157s	3207s	3014w	1550m	3353m	
				3353m				
Cl_2 Cd(HZI)(3-amp) ₂ [1683s	1606s	3157m	3197s	3070w	1552m	3352m	
				3362m				
Cl ₂]Cd(HZI)(4-amp) ₂ [1685s	1587s	3157s	3197m	3055w	1552m	3353m	
				3360m				

s = strong, m= medium, w= weak , Ar.= Aromatic

NMR Spectra

¹H-NMR of (HZI) ligand

The 1 H-NMR spectrum of (HZI) ligand in DMSO-d⁶ showed the protons H_{a} and H_{d} as doublet of doublets at δ =7.36ppm and δ =6.87ppm with coupling constant (3 J_{Ha-Hb}=8.03Hz) and (3 J_{Hd-Hc}=7.87Hz) respectively. Whereas the proton in position H_{b} and H_{c} appeared as doublet of triplets at δ =6.98ppm and δ =7.16ppm with

coupling constant ($^3J_{Hb\text{-Hc}}$ =7.78 Hz) and the spectrum showed two doublet at δ =9.53ppm and δ =10.54ppm with coupling constant ($^2J_{H\text{-H}}$ =13.94 Hz) respectively attributed to each of (NH₂) proton [13], and showed a single signal at δ =10.67ppm attributed to the (NH) proton for isatin.

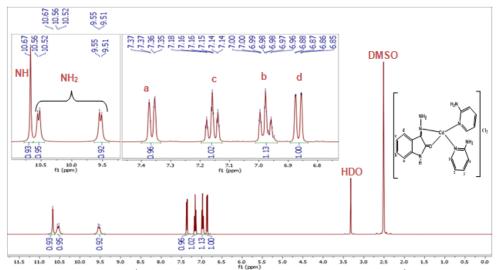


Fig. 3: ¹H-NMR spectrum of (HZI) ligand in DMSO-d⁶

¹³C-NMR of (HZI) ligand

The 13 C-NMR spectrum of the (HZI) ligand in DMSO-d⁶ showed a signal at δ C=136.48ppm which was attributed to the carbon (C=N) group, as well as a signal at δ C=140.48ppm assigned to the carbon of

benzene ring which was bonded to N pentagonal ring. The carbonyl group appeared at δC =162.91ppm, while the another peaks of the carbon atoms showed within δC =111.35-129.54ppm range[13].

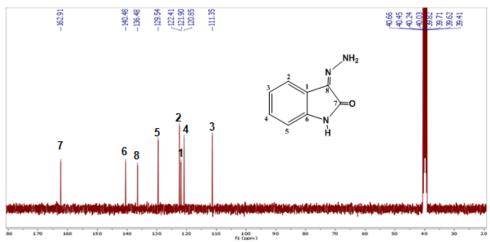


Fig.4: ¹³C-NMR spectrum of (HZI) Ligand in DMSO-d⁶

¹H-NMR of [Cd(HZI)(2-amp)₂]Cl₂ complex

The spectrum of $[Cd(HZI)(2-amp)_2]Cl_2$ complex showed a doublet signal at δH =6.86ppm with coupling constant (${}^3J_{Hd-Hc}$ =7.74Hz) due to the H_d proton, and a triplet of doublets at δH =6.97ppm with coupling constant (${}^3J_{Hb-Hc}$ =7.50Hz), (${}^4J_{Hb-Hd}$ =1.02Hz) attributed to proton H_b proton, another signal showed a triplet of doublets at δH =7.15ppm with coupling constant (${}^3J_{Hc-Hb}$ =7.61Hz), (${}^4J_{Hc-Ha}$ =1.31Hz) attributed to the H_c proton, the spectrum showed a doublet at δH =7.36ppm attributed to the H_a proton, with coupling constant (${}^3J_{Ha-Hb}$ =7.56Hz), the spectrum also showed two doublets at δH =9.53ppm and δH =10.54ppm with coupling constant (${}^2J_{H-H}$ =14.22 Hz) and (${}^2J_{H-H}$ =14.12 Hz) respectively attributed to each

of (NH_2) protons[13], and showed a single signal at δH =10.68ppm attributed to the (NH) proton for isatin

The spectrum showed the signals of protons of the 2-amp as a broad singlet at δH =5.99ppm attributed to the (NH₂) where the integration value confirms the presence of 4 protons of the two (NH₂) groups, and the protons of H4 and H2 showed a multiple at δH =6.48 ppm, and the H3 appeared a triplets of doublet at δH =7.38 ppm with coupling constant ($^3J_{H-H}$ =7.12Hz), ($^4J_{H-H}$ =1.97Hz), the spectrum showed a doublet of doublets at δH =7.90 ppm due to the H1 proton with coupling constant ($^3J_{H1-H2}$ =5.1Hz), ($^4J_{H1-H3}$ =1.08Hz).

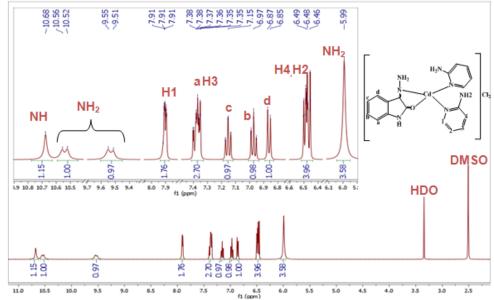


Fig. 5: ¹H-NMR spectrum of [Cd(HZI)(2-amp)₂]Cl₂ complex in DMSO-d⁶

¹³C-{¹H}NMR of [Cd(ZHI)(2-amp)₂]Cl₂ complex The ¹³C-{¹H}NMR spectrum of complex in DMSO-d⁶ showed a signal at δ C=139.39ppm due to the carbon of (N=C) group, and the δ C=149.39ppm carbon in position 5 in 2-amp and at δ C =159.79ppm

to the carbon position 1 in 2-amp, a signal at δ C=163.51 ppm attributed to the carbon of (C=O) group, while the another signals of the carbon atoms showed within δ C=137.63-109.95ppm range.

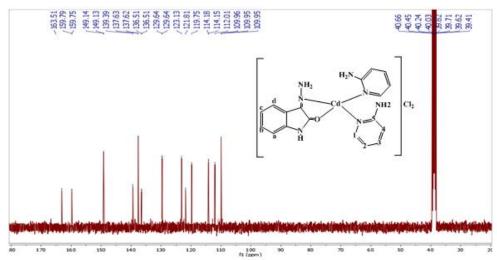


Fig. 6: ¹³C-NMR spectrum of [Cd(HZI)(2-amp)₂]Cl₂ complex in DMSO-d⁶

¹H-NMR of [Cd(HZI)(3-amp)₂]Cl₂ complex

The spectrum of complex showed a doublet of doublets at δH =6.86ppm with coupling constant ($^3J_{Hd-Hc}$ =7.83Hz), ($^4J_{Hd-Hb}$ =2.53 Hz) due to the H_d proton, and a triplet of doublets at δH =6.97ppm with coupling constant ($^3J_{Hb-Hc}$ =7.54Hz), ($^4J_{Hb-Hd}$ =2.72Hz) attributed to proton H_b proton, another signal showed as a triplet at δH =7.16ppm with coupling constant ($^3J_{Hc-Hb}$ =7.63Hz), attributed to the H_c proton, the spectrum showed a doublet at δH =7.36ppm with coupling constant ($^3J_{H1-H2}$ =8.51Hz), attributed to the H_a and H1 of 3-amp protons where the integration value confirms the presence of 3 protons, the spectrum also showed two doublets at δH =9.54ppm and δH =10.54ppm with coupling constant (2J_H

 $_{\rm H}$ =14.54 Hz) and ($^2{\rm J}_{\rm H\text{-}H}$ =14.84 Hz) respectively attributed to each of (NH₂) protons[13], and showed a single signal at $\delta{\rm H}$ =10.68ppm attributed to the (NH) proton for isatin.

The spectrum showed signals of 3-amp as a broad singlet at δH =5.13ppm attributed to the (NH₂) where the integration value confirms the presence of 4 protons of the two (NH₂) groups, and showed a doublet at δH =6.44 ppm attributed to the H3 protons with coupling constant ($^3J_{H3-H2}$ =9.26 Hz), ($^4J_{H3-H1}$ =2.33 Hz), and a triplet at δH =6.57 ppm attributed to proton H2 with coupling constant ($^3J_{H2-H1}$ =8.13Hz), and a doublet at δH =7.90 ppm attributed to the H4 protons with coupling constant ($^4J_{H4-H1}$ =4.16 Hz)[16].

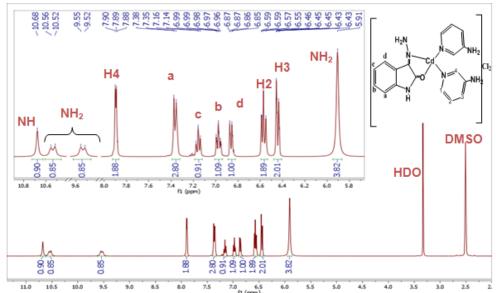


Fig. 7: ¹H-NMR spectrum of [Cd(HZI)(3-amp)₂]Cl₂ complex in DMSO-d⁶

¹³C-NMR of [Cd(HZI)(3-amp)₂]Cl₂ complex

The spectrum of complex showed a signal at δC =139.13ppm attributed to the carbon of benzene ring which was bonded to N pentagonal ring, the spectrum showed a signal at δC =149.39ppm attributed to the carbon Position 5 in 3-amp, signal at

 δC =160.17 ppm attributed to the carbon Position 5 in 3-amp, a signal at δC =163.25 ppm attributed to the carbon of (C=O) group, while the another signals of the carbon atoms showed within δC =108.66-127.51ppm range[16.

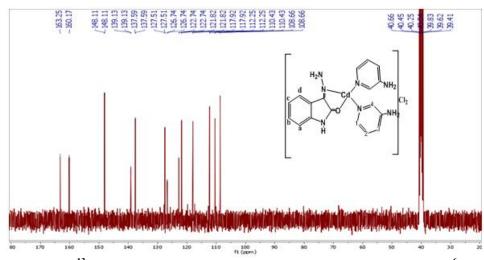


Fig. 8: ¹³C-NMR spectrum of [Cd(HZI)(3-amp)₂]Cl₂ complex in DMSO-d⁶

^{1}H -NMR spectrum of $[Cd(HZI)(4-amp)_{2}]Cl_{2}$ complex

The spectrum of complex showed a doublet at $\pmb{\delta}H=6.86pp$ with coupling constant ($^3J_{Hd\text{-Hc}}=7.66Hz$) due to the H_d proton, and a triplet at $\pmb{\delta}H=6.97ppm$ with coupling constant ($^3J_{Hb\text{-Hc}}=7.56Hz$) attributed to proton H_b proton, another signal showed a triplet of doublets at $\pmb{\delta}H=7.14ppm$ with coupling constant ($^3J_{Hc\text{-Hd}}=7.61Hz$) ($^4J_{Hc\text{-Ha}}=1.49Hz$), attributed to the H_c proton, the spectrum showed a doublet at $\pmb{\delta}H=7.36ppm$ attributed to the H_a with coupling constant ($^3J_{Ha\text{-Hb}}=6.88Hz$), the spectrum also showed two doublets at $\pmb{\delta}H=9.53ppm$ and $\pmb{\delta}H=10.54ppm$ with coupling constant ($^2J_{H\text{-H}}=13.95$ Hz) and ($^2J_{H\text{-H}}=10.00$

Hz) respectively attributed to each of (NH_2) protons[13], and showed a single signal at δH =10.68ppm attributed to the (NH) proton for isatin.

The spectrum showed signals of 4-amp as a broad singlet at δH =5.98ppm attributed to the (NH₂) where the integration value confirms the presence of 4 protons of the two (NH₂) groups, and showed a doublet of doublets at δH =6.45 ppm attributed to the H2 protons with coupling constant ($^{3}J_{H-H}$ =7.94) ($^{4}J_{H-H}$ =3.91Hz), and showed a doublet of doublets at δH =6.58 ppm attributed to the H1 protons with coupling constant ($^{3}J_{H-H}$ =8.80Hz) ($^{4}J_{H-H}$ =3.99).

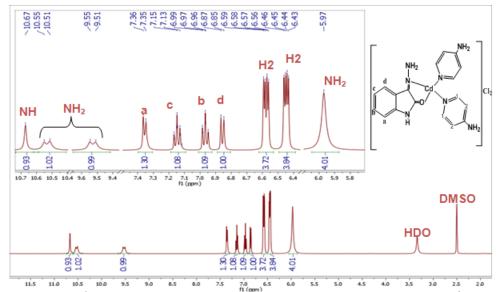


Fig. 9: ¹H-NMR spectrum of [Cd(HZI)(4-amp)₂]Cl₂ complex in DMSO-d⁶

$^{13}\text{C-}\{^1\text{H}\}\text{NMR Spectrum of }[\text{Cd}(\text{HZI})(4\text{-amp})_2]\text{Cl}_2$ complex

The spectrum showed a signal at δC =110.16 ppm due to the carbon of position 2 in 4-amp and a signal at δC =136.51ppm due to the carbon of (C=N) group, and a signal at δC =139.39 ppm attributed to the carbon of benzene ring which was bonded to N pentagonal ring, the spectrum showed a signal at

 δC =149.47ppm attributed to the carbon of position 1 in 4-amp and a signal at δC =153.97 ppm due to the carbon of position 3 in 4-amp, a signal at δC =163.51 ppm attributed to the carbon of (C=O) group, while the another signals of the carbon atoms showed within δC =112.01-129.64ppm range.

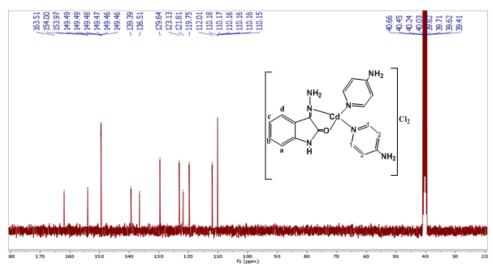
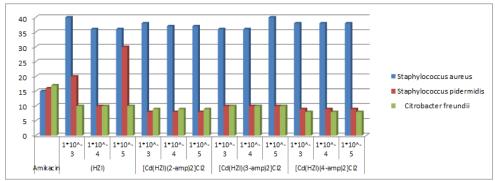


Fig. 10: ¹³C-NMR spectrum of [Cd(HZI)(4-amp)₂]Cl₂ complex in DMSO-d⁶

Biological activity study of the prepared compounds

The evaluation of biological activity of the prepared complexes against two bacterial types *Staphylococcus Epidermidis* and *Staphylococcus aureus* (gram positive) and *Citrobacer Freundii* (gram negative) by hole method in $(10^{-3}, 10^{-4}, 10^{-5} \text{ M})$ of solution of the

prepend complexes in DMSO-d⁶ [17-19] compared with Amikacin as standard antibiotic, All complexes showed high activity against *Staphylococcus aureus* more than amikacin, while the [Cd(HZI)(3-amp)₂]Cl₂ complex showed high activity against *Staphylococcus Epidermidis* and *Citrobacter freundii* more than another complexes.



Scheme (2) The biological activity of the prepared complexes aganist three types of bacteria speices.

Conclusions

A new tetrahedral complexes of the type $[Cd(HZI)(n-amp)_2]Cl_2$ (if n=2,3,4) when prepared by the react equal molar of $CdCl_2.2.5H_2O$ and HZI ligand with two moles of n-aminopyridine (n=2,3,4). The HZI ligand behaves as a bidentate chelating ligand thought the N atom of the azomethine group and O atom of carbonyl group, whereas the n-aminopyridine ligands

bonded as a monodentate through the N atom of heterocyclic ring.

And the prepared complexes showed a high biological activity against the *Staphylococcus aureus* mor than amikacin, while the [Cd(HZI)(3-amp)₂]Cl₂ complex showed activity against *Staphylococcus Epidermidis* and *Citrobacter freundii* compared with free ligand and Amikacin.

Table 3: Dimeler inhibition zone (in mm) of the prepend complexes in DMSO (DIZ) in (mm)

Compounds	Concentration	Staphylococcus	Staphylococcus	Citrobacter
		aureus	pidermidis	freundii
Amikacin	1*10^-5	15	16	17
(HZI)	1*10^-3	40	20	10
	1*10^-4	36	10	10
	1*10^-5	36	30	10
[Cd(HZI)(2-amp)2]Cl ₂	1*10^-3	38	8	9
	1*10^-4	37	8	9
	1*10^-5	37	8	9
[Cd(HZI)(3-amp) ₂]Cl ₂	1*10^-3	36	10	10
	1*10^-4	36	10	10
	1*10^-5	40	10	10
[Cd(HZI)(4-amp) ₂]Cl ₂	1*10^-3	38	9	8
	1*10^-4	38	9	8
	1*10^-5	38	9	8

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تحضير وتشخيص وتقييم الفعالية البايولوجية لبعض معقدات Cd(II) مع مزيج من ليكاندات -2هيدراز ونواندولين-2اون والامينات

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الملخص

تتضمن هذا البحث تحضير وتشخيص عدد من معقدات الكادميوم (II) مع ليكاند (3-hydrazonoindolin-2-one) وعدد من الليكاندات, أمينو بيربدين .

عند تفاعل مول واحد من ملح الفلز CdCl_{2.}2.5H₂O مع مول من (HZI) ومولين من n-أمينوبريدين (n:2,3,4) اتكونت معقدات رباعية السطوح ذات صيغة Cd(l_{2.}2.5H₂O) حيث يسلك الليكاند (HZI) سلوك ثنائي السن عن طريق ذرة نتروجين مجموعة الكاربونيل في حين يسلك n-amp سلوك أحادي السن من خلال ذرة نتروجين الحلقة غير المتجانسة .

شخصت المركبات المحضرة بواسطة التوصيلية المولارية وأطياف الاشعة تحت الحمراء وأطياف الرنين النووي المغناطيسي لنواة البروتون -H- NMR والكاربون IC-{1H}NMR والكاربون VMR

دُرست الفعالية البايولوجية للمعقدات المحضرة ضد نوعين من البكتريا الموجبة لصبغة كرام Staphylococcus aureus و كرم المعقدات المحضرة ضد نوعين من البكتريا الموجبة لصبغة كرام المحقدات المدروسة فعالية عالية اتجاه Staphylococcus و الخرى سالبة لصبغة كرام (Citrobacer Freundii ، اظهرت جميع المعقدات المدروسة فعالية اتجاه amikacin بينما ابدى المعقد (Cd(HZI)(3-amp)] ثاثير اكبر على النوعين Epidermidis و Epidermidis و Epidermidis بالمقارنة مع بقية المعقدات المدروسة.