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Preparation Metal Complexes of New Schiff Base Derived From 2-Aminoanthraquinone and 3-Acetyl Furan

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Abstract

In the present study, new metal complexes were synthesized using the reactive chemicals 2-amino anthraquinone and 3-acetyl furan to produce novel Schiff base ligands in molar ratios of 1:1 and 1:2 (metal to ligand) with the resultant complexes having the formulas [MLCl] and [ML_2Cl_2]. Several physicochemical techniques (Elemental analysis, molar conductivity, IR, UV-Vis spectroscopy and magnetic measurements) were used to characterize the complexes. Measurements of conductivity showed that none of the compounds were electrolytes. According to magnetic moment and electronic spectra, certain complexes have a tetrahedral geometry, while others have an octahedral geometry. In both circumstances, the ligand functions as a bidentate chelating ligand. Furthermore, biological assays reveal that several of the newly synthesized complexes demonstrate antibacterial properties, effectively inhibiting the proliferation of specific bacterial strains.

Keywords: 2-Aminoanthraquinone, Metal ions, 3-Acetyl Furan, Biological activity.

تحضير معقدات فلزية لقاعدة شف جديدة مشتقة من 2-امينو انثراكوينون و3-اسيتايل فيوران

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الخلاصة

في الدراسة الحالية، تم تحضير معقدات فلزية جديدة باستخدام مركبات كيميائية فعالة من 2-امينو انثراكوينون و3-اسيتايل فيوران لإنتاج ليكاندات قاعدة شف جديدة بنسب مولية (1:1) و(2:1) (فلز الى ليكاند) لإنتاج معقدات التي لها الصيغ [$MLCl_2$] و[ML_2Cl_2] . تم استخدام العديد من التقنيات الفيزيائية الكيميائية (التحليل العنصري، التوصيل المولي، مطيافية الأشعة تحت الحمراء، مطيافية الأشعة فوق البنفسجية والمرئية والقياسات المغناطيسية) لتوصيف المركبات. أظهرت قياسات التوصيل أن أياً من المركبات ليس إلكتروليتي. وفقًا للعزم المغناطيسي والأطياف الإلكترونية، فإن بعض المركبات لها هندسة رباعية السطوح، في حين أن البعض الآخر له هندسة ثماني السطوح. في كلتا الحالتين، يعمل الليكاند كليكاند ثنائي السن. وعلاوة على ذلك، تكشف الاختبارات البيولوجية أن العديد من المعقدات المحضرة حديثًا تظهر صفات مضادة للبكتيريا، وتثبط بشكل فعال انتشار سلالات بكتيرية محددة.

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1. Introduction:

In coordination chemistry, Schiff bases are compounds formed when primary amines react with ketones or aldehydes [1] which are a class of compounds containing an azomethine group (-C=N-) as functional group [2, 3]. Schiff bases have a variety of functions, including antibacterial properties, anti-inflammatory qualities, analgesic properties, anticancer properties, antioxidant properties, anti-tubercular properties and local painkillers. It is important in DNA replication, cardiovascular properties and their use as fluorination agents in industrial applications [4-18].

Anthraquinones [9, 10-anthracenediones] are widely used in chemo sensors [19]. These compounds represent a significant category of chemical substances, notable for their remarkable biological effects observed in living organisms. In particular, their ability to combat fungal infections stands out among their various properties, beside of, anthraquinones have properties as antioxidants, antimalarial, antibacterial, anti-leukemic, hypotensive activity, antitumor, anti-inflammatory, antimicrobial, antiviral and anti-HIV properties [20-26].

Many amino anthraquinones have a significant role in preventing the spread of cancer cells [27]. Anthraquinones are a sizable group of naturally occurring substances that are found in foods and are utilized in the pulp & paper, dye, and textile industries [28].
Kadhim *et al*. [29] examined the structure by combining 4,5-diphenyl imidazole with Hg(II) and Zn(II) metals. The coupling component was used to prepare the ligand from the amine group to synthesis of azo derivative from benzylamine. According to atomic absorption and conductivity measurements, a new azo Schiff ligand was identified and proposed. The three complexes have octahedral geometrical structures [29].

Khan *et al*. synthesized novel heterocyclic derivatives of 1-amino anthraquinone that contain 2-aminobenzothiazole. The following characterization techniques: FTIR, ^{1}H NMR and ^{13}CNMR were utilized to prove the structure of synthesized compounds.
L_2= 5-nitroindazole is used to synthesis a new series of hetero-binuclear complexes of the form $[M_1L_1Cl_2M_2(L_2)_2]$, which are then characterized using IR, UV-visible, and EPR spectroscopy. Magnetic moments and molar conductance in dimethylformamide(DMF) revealed that the compounds were covalent. A square planar geometry is recommended for M_2 and an octahedral geometry is suggested for M1 for the binuclear complexes [31].

The present study aims to synthesis new Schiff bases of 2-aminoanthraquinone and 3-acetyl furan and their complexes with new metal ions, as well as to examine their biological activity.

2. Experimental:

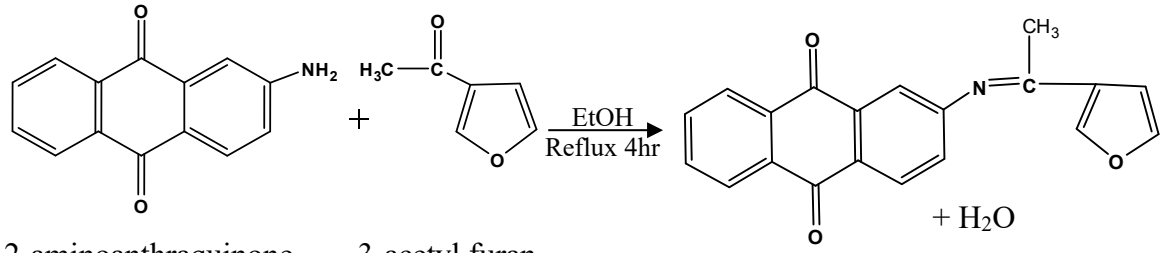
2.1. Physical measurement:

FTIR spectra of the ligand and its associated complexes were obtained using a Bruker Fourier Transform Infrared (FT-IR) spectrophotometer. The spectra were recorded in the range of 4000-200 cm^{-1}, with samples prepared as potassium bromide (KBr) pellets. Conductivity meter was employed to measure molar conductance of the complexes in a 10^{-3}M solution of DMF at room temperature. For 10^{-3} M solutions of the complexes in DMF, electronic spectra using a Shimadzu UV-visible spectrophotometer UV-160. The magnetic susceptibility of the complexes was determined using the Faraday method at 25°C on a solid sample, utilizing a Bruker BM6 apparatus. Melting points were determined using an Electrothermal 9300 instrument. The metals contents of the complexes (Ni, Cu, Zn) were estimated volumetrically using standard solution of EDTA and suitable indicator [32]. Cobalt was determined spectrophotometry using UV-visible [33], however, Cadmium and manganese were

determined using atomic absorption spectroscopy (PYE Unicam Spg, Holland). Mohr's technique was used to determine the chloride content [32]. Finally, the synthesized complexes and ligands were analyzed using C.H.N. microanalysis at the laboratories of Ordu University in Ordu, Turkey.

2.2. Preparation of ligand

A 20 ml ethanolic solution contains 2-amino anthraquinone (0.01 mole, 2.2 gm) was mixed with solution of 3-acetyl furan (0.01 mole, 1.10 gm) to create the Schiff base ligand. The reaction mixture underwent reflux for a duration of four hours. Subsequently, the product was isolated through filtration, washed with ethanol, and then treated with ether. The resulting substance was purified by recrystallization and left to dry naturally in air. The synthetic route for this ligand is illustrated in Scheme 1 below:



2-aminoanthraquinone 3-acetyl furan Schiff base

Scheme 1: The synthesis step of ligand.

2.3. Complexes preparation

Preparation of [$MLCl_2$] (1-6) complexes

A 20 ml ethanolic solution of Schiff base (1 mmole, 0.320 gm) was mixed with solution of $MnCl_2.4H_2O$ (1 mmole, 0.19 gm). The combination was then refluxed for two hours while being continuously stirred. After allowing the mixture to cool, the solid product that has precipitated out was isolated via filtration with ice-cold ethanol and ether. The solid was then allowed to air dry. Subsequently, the synthesis of Co(II), Ni(II), Cu(II), Zn(II), and Cd(II) complexes was conducted using the similar process. The process used to prepare the subsequent complexes (2–6) is detailed in Table (1).

Table 1: The weights of metal salt and ligand used ([$MLCl_2$] (1-6) complexes).

Salt	Wight (gm) of metal salt	Weight (gm) of ligand
$MnCl_2.4H_2O$	0.197	0.320
$CoCl_2.6H_2O$	0.237	0.320
$NiCl_2.6H_2O$	0. 238	0.320
$CuCl_2.2H_2O$	0.17	0.320
$ZnCl_2$	0.137	0.320
$CdCl_2.2(1/2)H_2O$	0.228	0.320

Preparation of [ML_2Cl_2] for (7–12) complexes

A solution of $MnCl_2.4H_2O$, (1 mmole, 0.197 g) in ethanol was mixed with Schiff base ligand 2-amino anthraquinone (2 mmole, 0.64 g). The mixture was refluxed for two hours while being stirred frequently. After that, the mixture was filtered out with ethanol and diethyl ether as shown in (Table 2).

Table 2: The weights of metal salt and ligand used ([ML_2Cl_2] (7-12) complexes).

Salt	Wight (gm) of metal salt	Weight (gm) of ligand
$MnCl_2.4H_2O$	0.197	0.640
$CoCl_2.6H_2O$	0.237	0.640
$NiCl_2.6H_2O$	0. 238	0.640
$CuCl_2.2H_2O$	0.17	0.640
$ZnCl_2$	0.137	0.640
$CdCl_2.2(1/2)H_2O$	0.228	0.640

2.4. Biological Study

Preliminary biological screening of the synthesized compounds against microorganisms, specifically Staphylococcus aureus and Escherichia coli, has been conducted using nutrient agar medium. In aseptic settings, 100 µL/ml DMF in opposition to microorganisms were spread onto agar plates by pouring the sterile agar into Petri dishes [34]. To determine a microorganism's susceptibility to antimicrobial compounds, assay plates were incubated at 37°C for 24 hours.

3. Results and discussion

The direct reaction of Schiff base with metals ions in ethanol produces complexes of the types [$MLCl_2$] and [ML_2Cl_2]. The synthesized complexes are solid compounds with distinct colors. The elemental analysis data for these newly formed complexes are presented in Table 3. The molar conductance of the formed complexes was within the range of (10-22)($ohm^{-1}cm^2mol^{-1}$) and showed that they were all nonelectrolytes [35]. Table 3 presents several characteristics of the ligand (L) and its complexes.

Table3: The analytical data obtained from the synthesized compounds.

No.	Compounds	Colour	M.P. (° C)	Yield (%)	Molar condctance $\Lambda_{M.}$ in DMF ($\Omega^{-1}cm^2mol^{-1}$)	M% .Found, (Calculated)	Cl% .Found, (Calculated)	Found (calc.)%		
								C..	.H.	.N.
L	$C_{20}H_{13}O_3N$	Brown	222-225	81.92	---	---	---	76.50 (76.19)	4.17 (4.12)	4.63 (4.44)
1	[$MnLCl_2$]	Brown	253-255	79.09	14	12.51 (12.45)	16.15 (16.10)	54.81 (54.43)	2.87 (2.94)	3.64 (3.17)
2	[$CoLCl_2$]	Green	261-263	85.85	19	13.32 (13.24)	16.04 (15.95)	53.78 (53.94)	2.44 (2.92)	3.02 (3.14)
3	[$NiLCl_2$]	Red-Purple	258-260	71.32	13	13.28 (13.20)	15.88 (15.96)	53.67 (53.96)	2.39 (2.92)	3.06 (3.14)
4	[$CuLCl_2$]	Brown	266-268	61.92	15	14.20 (14.13)	15.88 (15.79)	53.23 (53.38)	2.81 (2.89)	3.04 (3.11)
5	[$ZnLCl_2$]	Brown	268-270	66.81	10	14.36 (14.48)	15.64 (16.72)	52.90 (53.17)	2.94 (2.88)	3.57 (3.10)
6	[$CdLCl_2$]	Red	263-265	77.69	22	22.48 (22.55)	14.30 (14.24)	48.29 (48.15)	2.48 (2.60)	2.75 (2.80)
7	[MnL_2Cl_2]	Brown	251-253	73.40	18	7.33 (7.26)	9.27 (9.39)	31.86 (31.74)	1.98 (1.71)	1.95 (1.85)
8	[CoL_2Cl_2]	Dark Green	260-262	79.03	13	7.82 (7.75)	9.41 (9.34)	31.37 (31.58)	1.89 (1.71)	1.78 (1.84)
9	[NiL_2Cl_2]	Red	268-270	69.78	18	7.63 (7.72)	9.48 (9.34)	31.67 (31.59)	1.93 (1.71)	1.97 (1.84)
10	[CuL_2Cl_2]	Brown	257-259	68.97	20	8.36 (8.31)	9.39 (9.28)	31.07 (31.39)	1.58 (1.70)	1.74 (1.83)
11	[ZnL_2Cl_2]	Orang - Brown	266-268	67.42	17	8.61 (8.52)	9.15 (9.26)	31.11 (31.31)	1.76 (1.69)	1.90 (1.82)
12	[CdL_2Cl_2]	Purple	269-271	73.33	12	13.73 (13.81)	8.80 (8.72)	29.43 (29.50)	1.48 (1.59)	1.80 (1.72)

3.1. IR Spectra

The characteristics bands of synthesized complexes are presented in the Table (4). In the ligand spectra, absorption bands were observed at 1643 cm^{-1} and 1725 cm^{-1}, which were assigned to the υ(C=N) and υ(C=O) vibrational stretches, respectively. Which shifts bands towards lower values (1599-1620) cm^{-1} and (1685-1708) cm^{-1} and that indicating for the nitrogen atom in azomethine group(-C=N-) [36, 37] and the oxygen atom of carbonyl are coordinated with metal ion [38, 39]. The starching vibration for (C-O-C) for the furan in ligand spectra at (1153-1157) cm^{-1} and when formation the complexes appear the band in the same site, that indicating it is not involved in the coordination between oxygen atom for furan group with the metal ions and that agreement with others papers [36].

The region showed the appearance of new bands that were absent from the produced ligand's spectra (459-481) cm^{-1}, (415-427) cm^{-1} which were attributed to *υ*(M-O), and *υ*(M-N.) indicating that the ligand coordinated through azomethine nitrogen and carbonyl oxygen to the metal ions [36, 41, 42].

Additionally, the IR spectra of all complexes exhibit extra bands in the range of 333-341 cm^{-1}, which may be attributed to the υ(M–Cl) vibrations [43, 44].

Table 4: The IR data obtained from the synthesized complexes

No.	Compounds.	υ(C=N),.	υ(C=O)	υ(C-O-C)	υ(M–Cl)	υ(M–N)	υ(M–O)
	(L)	1643	1725	1155	---	---	---
1	$[MnLCl_2]$	1608	1705	1153	335	418	467
2	$[CoLCl_2]$	1612	1691	1155	340	427	471
3	$[NiLCl_2]$	1605	1703	1157	336	421	459
4	$[CuLCl_2]$	1607	1689	1157	334	415	469
5	$[ZnLCl_2]$	1599	1685	1156	336	425	464
6	$[CdLCl_2]$	1620	1694	1154	333	416	460
7	$[MnL_2Cl_2]$	1611	1708	1156	335	419	473
8	$[CoL_2Cl_2]$	1607	1690	1154	339	420	481
9	$[NiL_2Cl_2]$	1603	1697	1156	341	423	469
10	$[CuL_2Cl_2]$	1609	1702	1155	338	427	480
11	$[ZnL_2Cl_2]$	1610	1693	1157	336	426	475
12	$[CdL_2Cl_2]$	1605	1700	1156	337	417	463

3.2. Electronic spectra

The observed shifts in these spectral bands to either lower or higher frequencies in the complex spectra provide evidence for the coordination between the ligand and the metal ion. The electronic spectra of the showed bands in the area (33609-36528) cm^{-1} and at (25753) cm^{-1} as shown in Table5, which can be assigned to (π→ π*) and (n→π*) transition of nitrogen of the azomethine groups.

Mn(II) complexes with (1) tetrahedral and (7) octahedral geometry did not show any prominent absorption that could be attributed to the d-d transition (Table 5), however, they did present any band which can be attributed to the charge transfer transition. The d-d transition in Mn(II) octahedral complexes is doubly forbidden from the ^6A_1g. towards quarter terms $^4A_1g(G)$; $^4Eg(E)$; $^4T_2g(G)$; $^4T_1g(G)$ are hidden by the intra ligand transition and exhibit relatively low intensity, and in tetrahedral environment. These transitions are approximately 100 times stronger than previous ones; as a result, the structure of the prepared Mn(II)

complexes (1 and 7) was hypothesized using information from other measurements, including metal content and IR spectra [45].

For tetrahedral cobalt (II), the complex (2) is given a band in the visible region (14899 cm^{-1}) assigned to the transition electron $^4A_2$ (F) → $^4T_1$ (P) (υ_3). While the other two bands υ_1 and υ_2 (due to $^4A_2$ (F) → $^4T_2$ (F) and $^4A_2$ (F) →$^4T_1$ (F) transition were absence because the fall below the limit of our instrument [46], and band at (26659-31773 cm^{-1}) which can be attributed to the charge transferee transition which also supported a tetrahedral arrangement around Co ion (II) [47] (Table 5).

Cobalt (II) complex (8) is shown three bands at (10741, 14890) cm^{-1} which were attributed to transitions ^4T_1g (F) →^4T_2g (F) and ^4T_1g (F) →^4A_2g (F) while the third band (20299 cm^{-1}) could be due to charge transfer transition. These results demonstrated that this complex's geometry is octahedral [48].

Ni(II) complex (3) showed a strong band at (13983) cm^{-1} region assignable to the $^3T_1$ (F) → $^3T_1$ (P) υ3 as d-d transition in tetrahedral geometry [49] and the band at (24431)cm^{-1}which could be attributed to charge transfer transition however, the band at (37170) cm^{-1}intra ligand. The position of the bands suggested that these complexes have a tetrahedral geometry. Meanwhile, the Ni(II) complex (9) exhibited three transition bands in the 10378 cm-1, 15469 cm-1, and 28317 cm-1 regions, which could be attributed to electronic transitions. ^3A_2g (F) →^3T_2g (F) and ^3A_2g (F) →^3T_1g (F) and ^3A_2g (F)→^3T_1g (P) [50, 51]. These bands are in accordance with the complex's octahedral geometry.

The copper (II) complex (4) display a broad band at (10751 cm^{-1}) due to $^2T_2$ → ^2E which indicate that the complex has tetrahedral structure, and other transition at (22680-32361 cm^{-1}) assigned to the charge transferee transition supporting tetrahedral geometry of the Cu(II) complexes[52].

Cu(II) complexes (10) also showed a broad band in the region 13183cm^{-1} indicated that ^2B_1g→ ^2B_2g, point out the octahedral geometry while other transition (33392 cm^{-1}) could be due to intra ligand[51] .

3.3. Magnetic Measurements

Table 5 presents the magnetic moment values for the complexes, measured at 25°C. The data includes information for the Mn(II) complex. Complex (1) exhibited at (5.618) B.M, which is typical of a tetrahedral, whereas complex (7) has a magnetic value of (5.851) BM. Which was attributed to high spin state octahedral geometry [37].

The Co(II) complexes (2) possess value of (4.22) B.M which is corresponding to tetrahedral geometry[48], while Co(II) complex (8) showed value of (5.198) B.M. and this high value is due to the orbital contribution in high spin octahedral geometry[53].

Ni(II) complex (3) has a (3.57) B.M and this value is typical of tetrahedral[54] environment around the nickel. Complex (9) had (3.177) B.M may be giving octahedral geometry [55].

The Cu(II) complexes (4, 10) have magnetic moment (1.51, 1.71) B.M. These findings are consistent with the complexes having one unpaired electron, which aligns with tetrahedral and octahedral geometries, respectively [56].

The complexes of both Zn(II) and Cd(II) (5, 6, 11, and 12) showed that they have no unpaired electrons and are characterized by an intense band at (28739-32482 cm^{-1}), which can be attributed to the charge transfer transition[57] from L to M and π to π^* transitions. However, upon complex formation, these bands may be shifted to other values. These results provide additional proof that the ligands and metal ions were coordinated [58].

Table 5: Presents the electronic and magnetic spectra of synthesized complexes

No.	Compounds	μeff.(B.M.)	Geometry	Absorption region cm^{-1}
	L	---	---	25753(n→ π^*) (λ_{max} 320 nm), 33609-36528 ($\pi \rightarrow \pi^*$)(λ_{max} 265 nm)
1	$[MnLCl_2]$	5.618	Td	20063,25790,32298
2	$[CoLCl_2]$	4.220	Td	14899, 26659, 31773
3	$[NiLCl_2]$	3.631	Td	13983, 24431, 37170
4	$[CuLCl_2]$	2.060	Td	10751, 22680, 32361
5	$[ZnLCl_2]$	(dia)	Td	28739, 30980
6	$[CdLCl_2]$	(dia)	Td	29742, 31339
7	$[MnL_2Cl_2]$	5.851	Oh	22019, 26631, 31342
8	$[CoL_2Cl_2]$	5.198	Oh	10741, 14890, 20299
9	$[NiL_2Cl_2]$	3.177	Oh	10378, 15469, 28317
10	$[CuL_2Cl_2]$	1.790	Oh	13183, 33392
11	$[ZnL_2Cl_2]$	(dia)	Oh	29872, 31154
12	$[CdL_2Cl_2]$	(dia)	Oh	29890,32482

Td = tetrahedral
Oh= Octahedral

From the results obtained and discussed in this paper, the suggested structure of complex No. (1-6) was proposed as in Fig. 1 and structure of complex No. (7-12) was proposed as in Fig. 2.

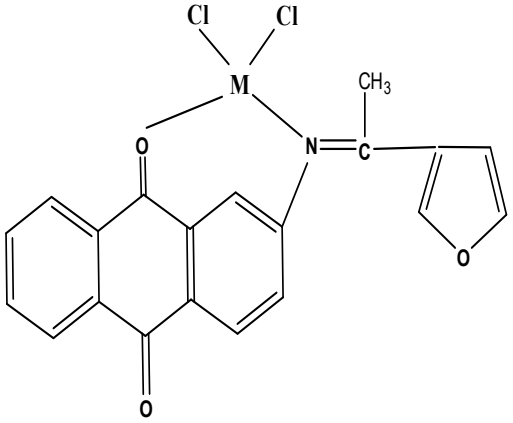


Figure 1: The suggested structure of complex No. (1-6).

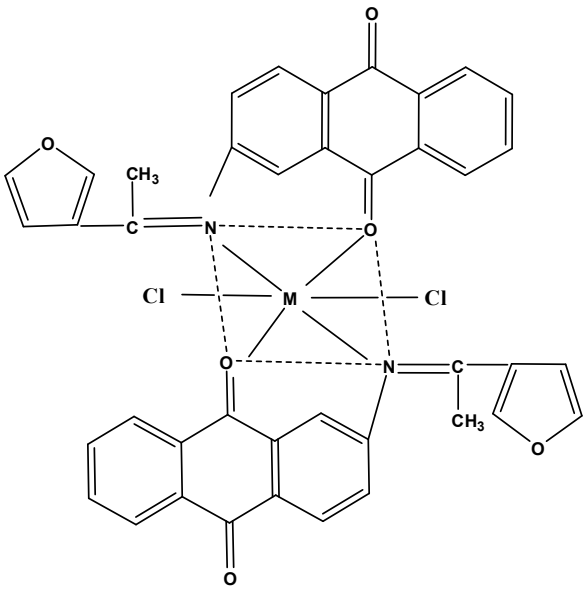


Figure 2: The suggested structure of complex No. (7-12).

3.4. Biological activity

Staph. aureus and *E. coli* organism have been studied by the disk diffusion method [59] as shown in Table (6). The results revealed that the $[CuLCl_2]$ and $[ZnLCl_2]$ complexes exhibited the highest antibacterial activity (Fig. 3), this may be due to the interference of these types of complexes with the growth enzymes of bacteria and inhibition of their growth.

Table 6: The biological activities of synthesized complexes

Comp.	Empirical formula	*Staph. aureus*	*E. coli*
		Zone of inhibition (mm)	Zone of inhibition (mm)
1	$[MnLCl_2]$	2	5
2	$[CoLCl_2]$	2	2
3	$[NiLCl_2]$	4	3
4	$[CuLCl_2]$	9	8
5	$[ZnLCl_2]$	12	4
6	$[CdLCl_2]$	11	6
7	$[MnL_2Cl_2]$	4	3
8	$[CoL_2Cl_2]$	2	5
9	$[NiL_2Cl_2]$	3	4
10	$[CuL_2Cl_2]$	10	7
11	$[ZnL_2Cl_2]$	11	2
12	$[CdL_2Cl_2]$	9	7

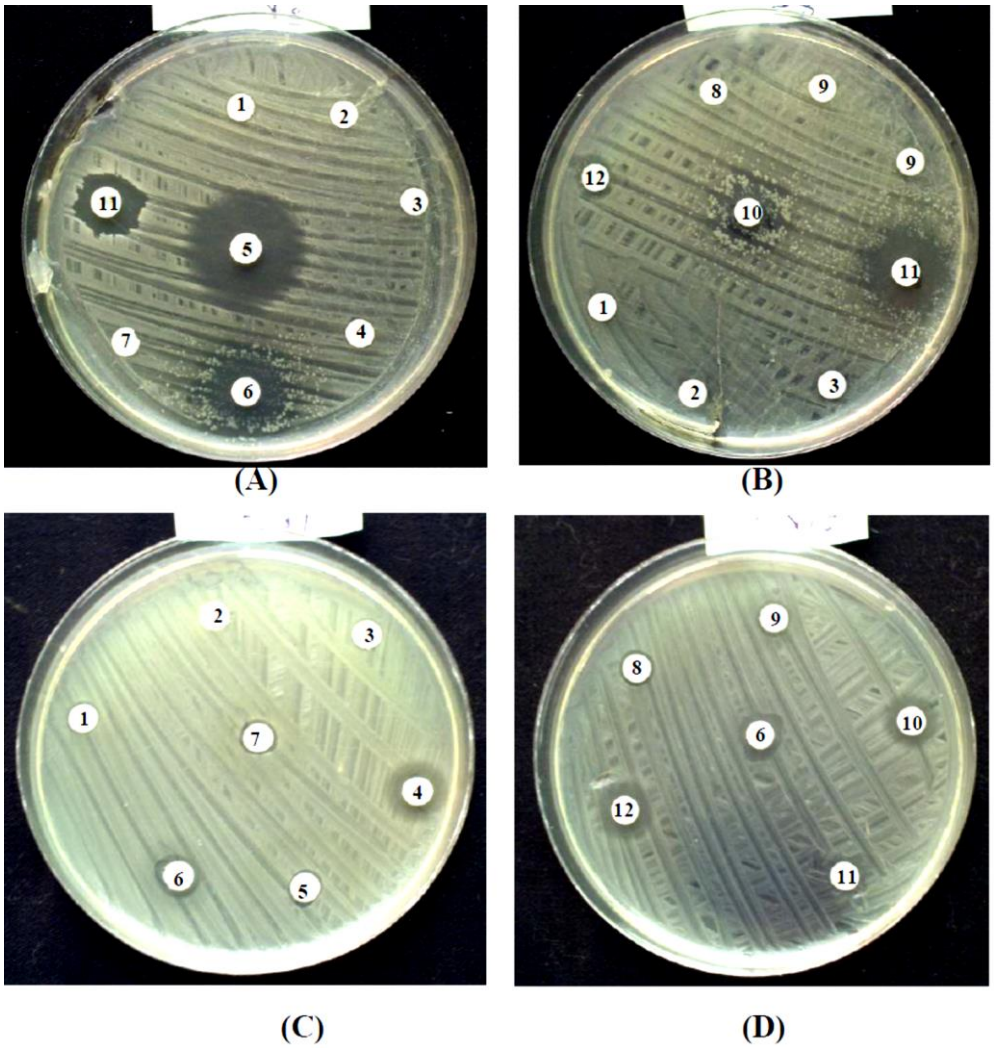


Figure 3: The *Staph* (A, B), *E.coli* (C, D).

Conclusion

Produce novel Schiff base ligands were synthesized using 2-amino anthraquinone and 3-acetyl furan having the formulas [MLCl] and [ML_2Cl_2]. Physicochemical analysis indicates that some of the complexes adopt a tetrahedral structure (complex No. (1-6)), while others exhibit an octahedral configuration (complex No. (7-12)), in these structures, the ligand acts as a bidentate chelating agent. Further biological assessments demonstrated that select complexes synthesized ([$CuLCl_2$], [$ZnLCl_2$]) possessed antimicrobial properties against certain types of bacteria, so it is possible to take prepared complexes with a high effect and use them to treat bacterial diseases.

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