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SYNTHESIS, CHARACTERIZATION AND STUDY THE ANTIBACTERIAL ACTIVITY OF SOME DISUBSTUTITED-1,3,4-OXADIAZOLE DERIVATIVES

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ABSTRACT:

The purpose of this study is to determine the useful of Schiff bases derivatives containing (oxazepine, tetrazole) rings with biological activity which can be used as drug and antimicrobial, the present work is started from [Binary (2,5(4,'4-diaminophenyl) – 1,3,4 – oxadiazole]. A variety of Schiff bases and heterocyclic (oxazepine, tetrazole) have been synthesis, and confirm that structures by physical properties , FTIR , 1H-NMR, 13C-NMR, elemental analysis, [Microbial study against three type of bacteria (staphylococcus aurea and klebsiella pneumonia) and (Canadida albncans) fungi].All analyzation performed in center of consulatation University of Jordan.

Key words: Heterocyclic ring compounds, antibacterial activity, Schiff bases.

تحضير وتشخيص مع دراسة الفعالية البايولوجية لمشتقات ثنائية التعويض (4,3,1-اوكساديازول)

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الخلاصة:

تم من خلال البحث تحضير عدد من مشتقات قواعد شف التي تحتوي على حلقات الاوكساسفين والتترازول مع الفعالية البايولوجية والتي ممكن استخدامها كدواء ومضادات للبكتريا وابتداءا من ثنائي (5,2-4,4'-ثنائي امينوفنيل)-4,3,1-اوكساديازول)، وتم تحضيرحلقات غير متجانسة (الاوكساسفين والتترازول) لقواعد شيف مختلفة، وشخصت هذه المركبات باستخدام تقنيات FTIR و13C-NMR, 1H-NMR والتحليل الدقيق للعناصر (C.H.N) لبعض منها فضلا عن تعين الخواص الفيزياوية ودراسة الفعالية البايولوجية ضد ثلاثة انواع من البكتريا، وجميعها اجريت في المركز الاستشاري بالجامعة الاردني

الكلمات المفتاحية: المركبات الحلقية الغير متجانسة، الفعالية البايولوجية، قواعد شف.

INTRODUCTION

Improvement of Synthesis the route to extended used organic compounds ring using readily available reagent is one of the main, objective of organic preparation, nitrogen heterocyclic are of special interest, because they constitute an important class of physics products ,many of which exhibit useful biological energy, one-pot efficient synthesis of heterocyclic derivatives, many permit the advancing of novel therapies for the treatment of epilepsy, pain and other neurodegen disorder **(Weiam & Zafer, 2018)**.

The Schiff bases which bearing aryl group **(Salim & Syrd, 2018)**, or heterocyclic residues posses excellent biological activities **(Oday, 2018)**. This has attracted many researchers' attention in recent year. They have been reported to use as analgesic, anthelmintic, antitubercuier, plant growth regular, antiviral, antifungal and anticancer. Derivative like the other members (oxazepine, tetrazole), all the (1,3,4-oxadiazole) sequence have been large applied as therapeutic agent due to their anticonvulsant, vasorelaxant and anti-inflammatory properties **(Sanaa *et al.*, 2017; Zainab & Hasan, 2018)**.

(1,3,4-oxadiazole) nucleus is many candid and synthesis biological energy compounds continues contribute to the development of new, synthesis methodologies, to word this important rings. Heterocyclic Schiff bases derivatives ,as well as known, to posses an array of physiological activities, like anticancer, muscle relexant, hypnotic, anti-inflammatory **(Rajkumar *et al.*, 2018)**, diuretic and anti hypertertensive activities and used in pharmaceuticals, promoted by the observations of above mentioned heterocyclic ring. According to the mentioned facts, it was thought worth, while to synthesize new compounds via introductioning the two biologically active moieties (oxazepine, tetrazole) or other heterocyclic in frame work followed by their antimicrobial screening **(Rajkumar *et al.*, 2018)**.

MATERIALS AND METHODS

General Methode

Melting point were determined on Gallenkamp (melting point) apparatus and were uncorrected, on (SHIMADZU)/ FTIR 8300 spectrometer as KBr. disc, result were given in (cm^{-1}), ^{1}H-NMR and ^{13}C-NMR spectra were recorded at 200.13 and 50.32 MH_Z respectively, (DMSO-d_6) which recorded in part per million (ppm) down field from internal tetramethylsilane (TMS) (chemical shift in δ values). Elemental analysis, were run using a perkin-Elmer RE2400 (C.H.N) analyzer, ^{1}H-NMR, ^{13}C-NMR. All analyzation performed in center of consulatation university of Jordan.

Material

All the chemical, used were supplied by (Merk, Fluka and BDH) chemical, the solvent purified by distillation and dried with calcium chlorid.

Preparation Bis (2,5-(4,4'-diaminophenyl)- 1,3,4- oxadiazole[1] (Iman, 2016):

Mixture (0.02 mol) (4-amino benzoic acid) with (0.01 mol) hydrazine hydrate, (5 ml) poly phosphorus acid, refluxing gently at (100-125) ^{o}C until the solution turned dark brown.

The cold reaction mixture was neutralized with Sodium bicarbonate and the resulting solid was filtered, dried and recrystallized from (ethanol) to give the desired oxadiazole derivative, physical properties and analyzide in (table, 1).

Preparation Schiff bases [2-7] (Bushra, 2015):

Schiff bases were prepared from reaction (0.01 mol) compound [1] with (0.02 mol) different aromatic aldehyde, ketones in (15 ml) abs. ethanol and (1-2) drops snowy acetic acid. Mixture was refluxed for 4h, cooled; precipitate was obtained, and then recrystallized from suitable solvent.

Preparation oxazepine compounds [8-13] (Kalida & Mohmoud, 2017):

Schiff bases [2-7] (0.01 mol) with (0.02 mol) pathalic anhydride refluxing for 5h, in oil bath (60-65) ^{o}C with (15 ml) dry benzene, the solid product precipitate, then separated upon cooling was filtered off , recrystallized from suitable solvent,physical properties and analyzide in (table, 1).

Preparation tetrazole compounds [9-19] (Maryna & Volodymyer, 2017):

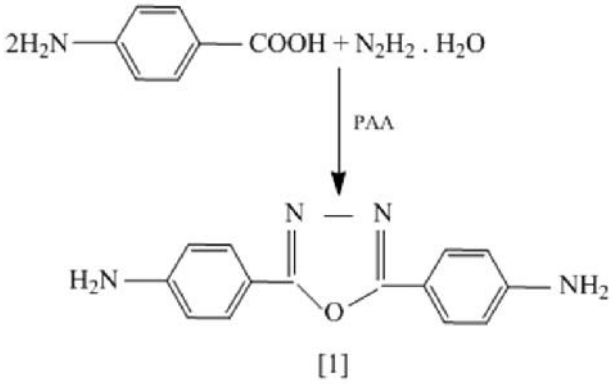
Schiff bases [2-7] (0.01 mol) with (0.02 mol) Sodium azide, the mixture refluxing in oil bath at (50-60) ^{o}C, (15 ml) THF. End reaction adjustment by (T.L.C), Solid product formed, filtered, recrystallize in suitable solvent, physical properties and analyzide in (table, 1).

Microbiological tests (Raed & Jame, 2018):

Agar (was added to (1L) of distilled water in suitable conical flask, stirring, heating until complete dissolve, then the flask was stoppered by cotton and the medium sterilized in an autoclave for (20 min) at (121) ^{o}C under pressure of (15 bound/inch). The medium was placed in petridishs about (20 ml) for each one and was left to cool and solidified. The studied bacteria and fungi were placed on the nutrient agar surface using the loop and by streaking processor then the discs saturated tested compound solutions. The samples were incubated for 24h at 37 ^{o}C.

RESULTS AND DISCUSSION

Considerable interest have been expressed in synthesis of Schiff bases in recent year due to their industrial, biological importance, based on bis [2,5 – (4,4'- diaminophenyl) – 1,3,4 – oxadiazole] [1]:

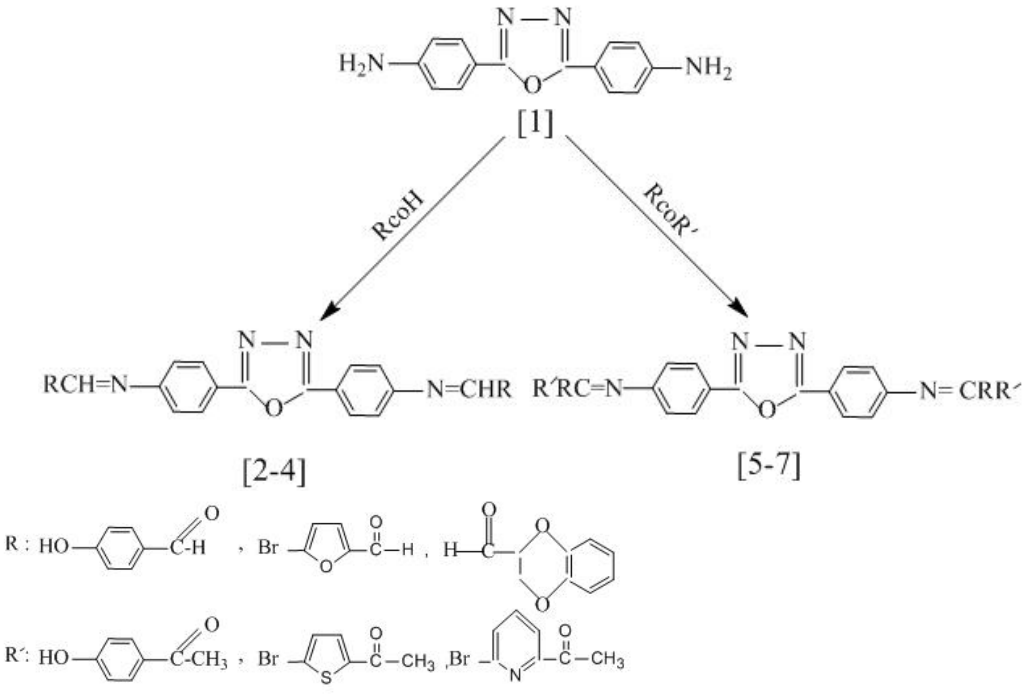


PAA = Polyphosphoric acid

Scheme -1-

The (IR) Spectrum showed the strong stretching vibration (3420-3375) due to (NH_2), (1200-1040) for (C-O-C), (830) for (1,4-disubst), (3080) for (Ar-H), (1420) for (C-N), (1593-1510) for (aromatic C=C), ^{1}H-NMR (DMSO-d_6) δ: (7.4-7.6) for (Ar-H), (8.7-9.3) due to (2H, NH_2); ^{13}C-NMR (DMSO-d6) δ: the four down field resonance (162.3, 163.18, 164.18, 164.65 and 165.94) are of benzene ring carbons at position (2,5) carbons bonded to the oxadiazole ring, (72.4-72.8) due to (C-O-C), (143.1-144.6) for (Ar-NH_2)[12]. (C.H.N) for compound [1] $C_{14}H_{12}N_4O$: [C, 66.7(67.7); H, 4.76 (5.45); N, 22.22 (23.10)].performed recorded in (table, 2-4).

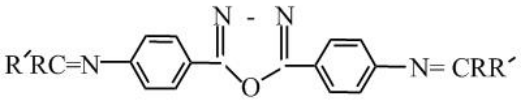
There for Schiff bases [2-7] prepared through condensation of the corresponding compound [1] with (aromatic aldehydes and ketons). The reaction proceeds by the nucliophilic attached of the nucleophilic nitrogen atom of the amine on the carbonyl group of aldehyde or keton with the loss of water molecular to give a stable compounds in good yield, which shown in Scheme -2-.



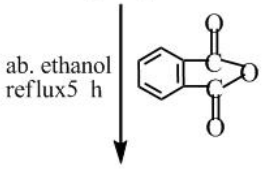
Scheme -2-

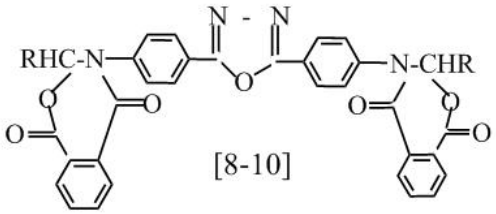
The (IR) spectrum showed the strong bands at (1602-1604) for (C=N) combined with dissolving of (NH_2), (2900-2845) due to (CH_3), (670) for (Br); for compounds (2,5), ^{1}H-NMR (DMSO-d_6) δ: (7.1-7.9) due to (Ar-H), (2.10-2.25) for (3H, CH_3), (10.1-10.6) for (H, OH); (^{13}C-NMR (DMSO-d_6) δ: (148.2-152.6) due to (C=N), (130.2-132.4) (aromatic carbons), (115.3-116.1) for (C, CH_3)[12]. (C.H,N) compound [2] $C_{28}H_{20}N_4O_3$ [C, 73.04 (74.02); H, 4.35 (5.32); N, 12.17 (13.15)] and for compound [5] $C_{30}H_{24}N_4O_3$: [C, 73.77 (74.80); H, 4.92 (5.92); N, 11.48 (12.46)]. Compounds [2-7] reacts with phthalic anhydride afforded (oxazepine) [8-13] compounds:

[2-4]

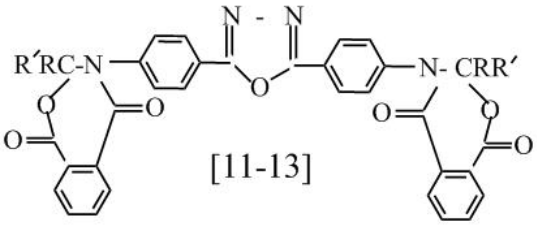


[5-7]

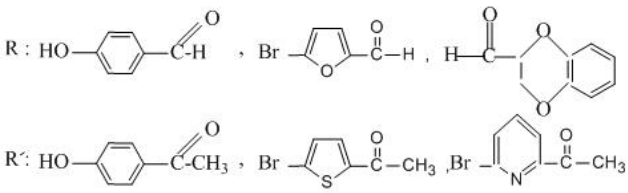




[8-10]



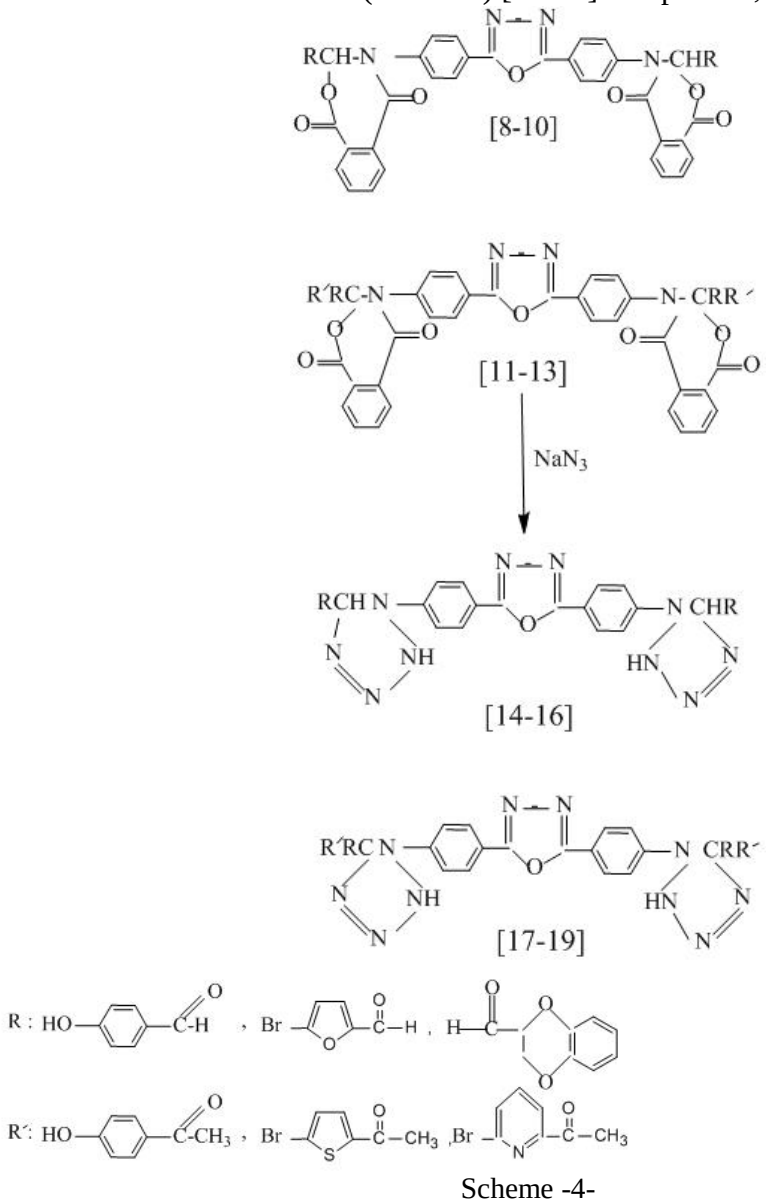
[11-13]



Scheme -3-

The (IR) spectrum (oxazepine) derivatives displayed the strong bands at (1755-1740) for (C=O) combined with dissolving for (C=N) , (1225-1200) for (C-O-C), (1280-1300) for (C=C), for compounds (8,11); ^{1}H-NMR (DMSO-d_6) δ: (7.5-8.1) due to (Ar-H), (10.6-11.1) to (H,OH), (2-2.1) for (3H, CH_3); ^{13}C-NMR (DMSO-d_6) δ: (160-164) due to (C=O), (148.2-151.1) due to

(aromatic carbons), (72.1-72.4) for (C-O-C), (26.3-26.4) due (C, CH_3)[12]. (C.H.N) for compound [8] $C_{44}H_{28}N_4O_9$ [C, 78.11 (79.10); H, 4.14 (5.10); N, 8.28 (9.25)], compound [11] $C_{46}H_{32}N_4O_9$ [C, 70.05 (71.03); H, 4.06 (5.04); N, 7.11 (8.10)]. Compounds [8-13] similarly reacts with Sodium azide afforded (tetrazole) [14-19] compounds, performed in (table, 2-4).



Scheme -4-

The (IR)[14-19] (tetazole) derivatives exhibit strong band(1450-1525) (N=N) combined with dissolving (C=O), (3230) for (NH), (1260-1280) for (C-N); ^{1}H-NMR (DMSO-d_6) δ: (7.6-8.8) due to (Ar-H), (9.4-9.7) due to (H-OH), δ: (1.9-2) for (3H, CH_3); ^{13}C-NMR (DMSO-d_6) δ: (9.1-9.2) due to (H, NH), (151-156) due to (aromatic carbons) (120-121) due to (N=N)[12]. (C.H.N) for compound [14] $C_{28}H_{22}N_{12}O_3$ [C, 58.54 (59.52); H, 3.83 (4.80); N, 29.27 (30.28)]; compound (17) $C_{30}H_{22}N_{10}O_3$: [C, 63.16 (64.10); H, 3.86 (4.86); N, 24.56 (25.50)].performed in (table, 2-4).

Microbial Study

The last part in this work involved evaluated of antimicrobial activity of the prepared Schiff bases derivatives against (staphylococcus aureus G^+Ve bacteria (klebsiella pneumonia) G^-Ve bacteria and (candida albicans) fungi. Inhibition zones caused by the various prepared compounds were determined and the results are listed in (table, 5).

The results showed that biological energy of the studied compounds depend on physics of substituents in molecular, compound (6,7,9,10,13,15,18,19) showed high biological activity due to the presence of (Br) and electron releasing substituents (CH_3) and (heterocyclic ring)[13]. Results at (table, 3) indicated that many (oxazepine) possess moderate to high biological activity against (G^+Ve) bacteria and this was due to the hydrophilic properties of these compounds and cell wall of (G^+Ve) bacteria, on other hand the molecules of the prepared (tetrazole) have hydrophilic properties and this in turn made these (G^-Ve) bacteria which posses complex (Lipo poly Soccharides) in their cell wall, Finally both (oxazepine, tetrazole) showed different biological activity against (*Candida albicans*), performed analyzide recorder in (table, 5).

Thus mostly compounds showed highly biologically activity while other moderate or weak activity.

Table (1): Physical properties of (1-19) compounds.

Comp No	Molecular formula	M-P C°	Colour	Yield%	Purification solvent	Comp No	Molecular formula	M-P C°	Colour	Yield%	Purification solvent
1	$C_{14}H_{12}N_4O$	198-200	brown	90	Ethanol	11	$C_{46}H_{32}N_4O_9$	Deco	light brown	70	Ethanol
2	$C_{28}H_{20}N_4O_3$	208-220	deep orang	75	Ethanol	12	$C_{42}H_{22}N_4O_7S_2Br_2$	Deco	coffee	75	Ethanol
3	$C_{24}H_{14}N_4OS_2Br_2$	135-137	coffee	65	Ethanol	13	C44H28N6O7Br2	Deco	coffee	75	Ethanol
4	$C_{32}H_{18}N_4O_5$	208-40	deep orang	60	Ethanol	14	$C_{18}H_{22}N_{10}O_3$	Deco	light brown	65	Methanol
5	$C_{48}H_{24}N_4O_3$	204-206	deep orang	60	Ethanol	15	$C_{19}H_{16}N_{10}S_2Br_2$	Deco	light brown	60	Methanol
6	$C_{26}H_{18}N_4OS_2Br_2$	164-166	coffee	75	Ethanol	16	$C_{32}H_{26}N_{10}O_5$	Deco	brown	55	Methanol
7	$C_{28}H_{20}N_4OBr_2$	150-152	coffee	55	Ethanol	17	$C_{30}H_{18}N_{10}O_3$	Deco	brown	55	Methanol
8	$C_{42}H_{12}N_4O_8$	200-202	brown	65	Ethanol	18	$C_{26}H_{20}N_{10}S_2Br_2$	Deco	light brown	60	Methanol
9	$C_{42}H_{18}N_4OS_2Br_2$	deco	light brown	80	Ethanol	19	$C_{28}H_{24}N_{10}Br_2$	Deco	light brown	65	Methanol
10	$C_{48}H_{32}N_{11}O_3$	deco	light brown	65	Ethanol	-	-	-	-	-	-

Dec: Decomposition

Table (2): IR Spectral of compound (1-19).

Comp No	$V_{(OH)}$	$V_{(C=N)}$	$V_{(C=C)}$	$V_{(CH3)}$	Others (V)	Comp No	$V_{(OH)}$	$V_{(C-N)}$	$V_{(C=C)}$	$V_{(CH3)}$	Others (V)
1	-	-	1597	-	NH2 (3420) (3375)	11	3280	1230	1590 1586	2980	C=O (1712) C-O-C (1230)
2	3400 3215	1602	1581	-	Ar-H(3010)	12	-	1255	1540 1535	2980 2880	C-Br (710) C=O (1722)
3	-	1610	1565	-	C-Br (670) C-S (1200)	13	-	1235	1550	2898	C=O (1730) C-Br (740)
4	-	1612	1598	2900	C-O-C (1255)	14	3300	1199	1593	-	NH (3210)

				2850			3200				N=N (1435-1555)
5	3350 3150	1610	1580	2985	C-O (1190)	15	-	1183	1597	-	NH (3300), N=N C-Br (705) (1445)
6	-	1613	1550	1875	C-Br (690) C-S (1250)	16	-	1180	1560	-	NH (3305) C-O-C (1200), N=N (1425)
7	-	1620	1572	2915	C-Br (695) C-N (1140)	17	3300	1200	1545	2910	NH (3295) C-Br (705), N=N (1420)
8	3200	-	1573 1492	-	C=O (1755), C-N (1140) C-O-C (1213)	18	-	1205	1538	2886	NH (3297) C-Br (699), N=N (1430)
9	-	-	1590 1570	-	C-Br (680) C=O (1739)	19	-	1210	1545	2940	NH (3200) N=N C-Br (678), (1422)
10	-	-	1538 1530	2885 2870	C=O (1722) C-O-C (1210)	-	-	-	-	-	-

Table (3): ^{1}H-NMR and ^{13}C-NMR Spectral data for some compounds**.**

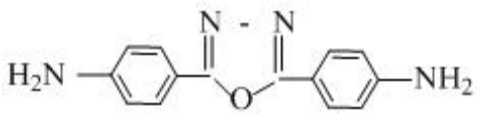
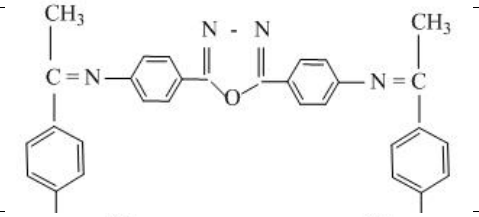
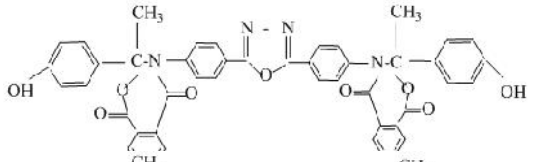
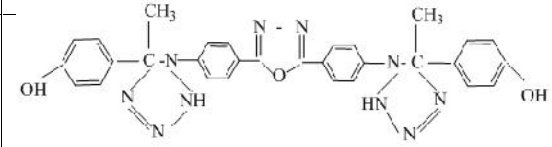
No	Structure	^{1}H-NMR/ data	^{13}C-NMR/ data
1		δ: 7.4-7.6 (Ar-H) δ: 8.7-9.3(2H, NH_2)	δ: 162.3-164.65 (benzene ring) δ: 72.4-72.8 (C-O-C) δ: 143.1-144.6(Ar-NH_2)
5		δ: 7.1-7.9(Ar-H) δ: 10.1-10. 6(H,OH) δ: 2.10-2.25 (3H,CH_3)	δ: 130.2-132.4 (aromatic carbons) δ: 148.2-151.6(C=N) δ: 155.3-116.1(C, CH_3)
8		δ: 7.5-8.1 (Ar-H) δ: 10.6-11.1(H,OH) δ: 2-2.1(3H,CH_3)	δ: 160-164 (C=O) δ: 148.1-151.1 (aromatic carbons) δ: 72.1-72.4 (C-O-C) δ: 26.3-26.4 (C, CH_3)
4		δ: 7.6-8.8(Ar-H) δ: 9.4-9.7(H,OH) δ: 1.9-2 (3H,CH_3)	δ: 9.1-9.2 (H,NH) δ: 151-156 (aromatic carbons) δ: 120-121 (N=N)

Table (4): (C.H.N) for Some Compounds.

No.	(C.H.N) calculated (found)			No.	(C.H.N) calculated (found)		
	%C	%H	%N		%C	%H	%N
1	66.7 (67.7)	4.76 (5.40)	22.22 (23.10)	8	78.11 (79.10)	4.14 (5.10)	8.28 (9.25)
2	73.04 (74.02)	4.35 (5.32)	12.17 (13.15)	11	70.05 (71.03)	4.06 (5.04)	7.11 (8.10)
5	73.77 (74.80)	4.92 (5.92)	11.48 (12.46)	14	58.54 (59.52)	3.83 (4.80)	29.27 (30.28)
				17	63.16 (64.10)	3.86 (4.86)	24.56 (25.50)

Table (5): Microbiological activities of compounds (2-19).

Comp No	*Staphylococcus aureus*	*Klebsiella pneumonia*	*Candida albicans* (fungi)	Zone inhibition in (mm)	Comp No	*Staphylococcs aureus*	*Klebsiella pneumonia*	*Candida albicans* (fungi)	Zone inhibition in (mm)
2	8	5	3	3	11	8	R	10	9
3	13	13	17	12	12	15	15	16	11
4	7	5	R	3	13	19	19	23	12
5	8	R	R	6	14	11	11	R	6
6	17	17	22	15	15	17	16	17	12
7	19	21	23	17	16	8	10	R	5
8	8	R	R	5	17	10	9	R	8
9	17	20	8	12	18	20	22	20	13
10	19	22	9	11	19	29	23	19	13

Key of symbols: R=Resistant; Inhibition zone < 6mm=inert.
Inhibition zone (6-9)mm=Slightly active; Inhibition zone (9-12)mm=moderately active; Inhibition zone > highly active.

CONCLUSION

Through this work we have succeeded synthesis heterocyclic rings (oxazepin, tetrazole) which give highly biological activity agreement with proposed structure. Most of the synthesized Schiff base derivatives were potential lead for industrial application and biological activity respectively, on the basis of observied results,it may concluded that the substitution favors the activity. The derivatives is known to be influenced to great extend by two aryl structure,i.e, Schiff base derivatives have been synthesis and evaluated for industrial application and biological activity.

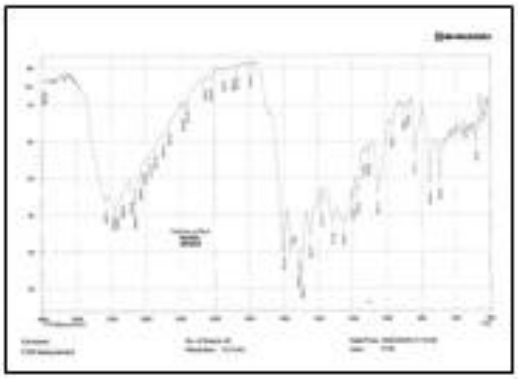


Fig. (1) FT-IR for compound (1)

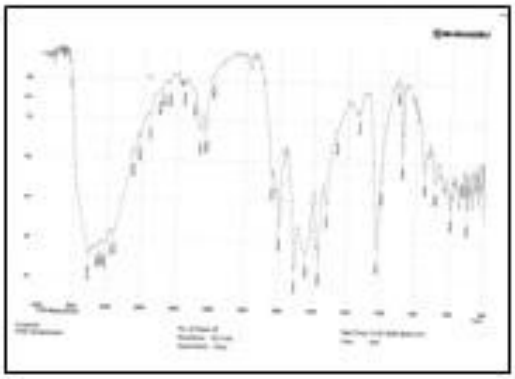


Fig. (2) FT-IR for compound (2)

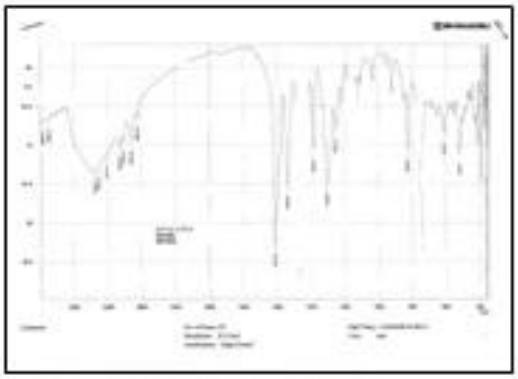


Fig. (3) FT-IR for compound (3)

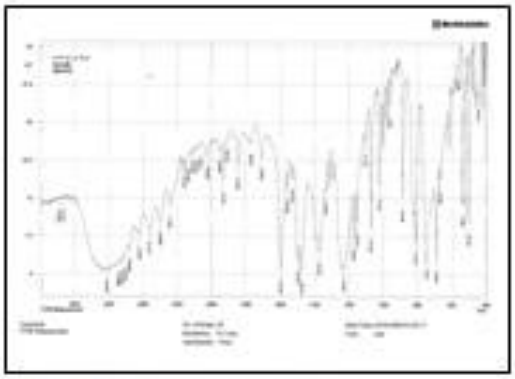


Fig. (4) FT-IR for compound (4)

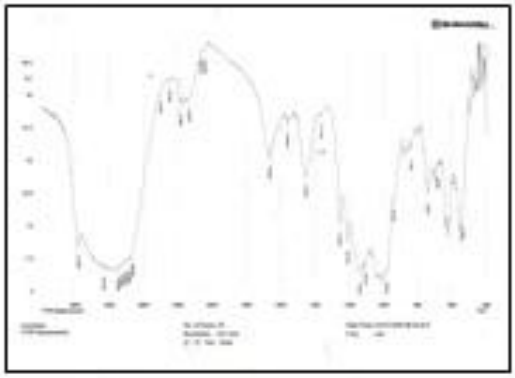
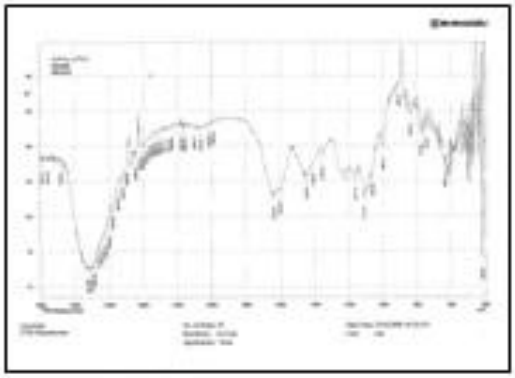


Fig. (5) FT-IR for compound (7)



Fig(6)FT-I R for compoun

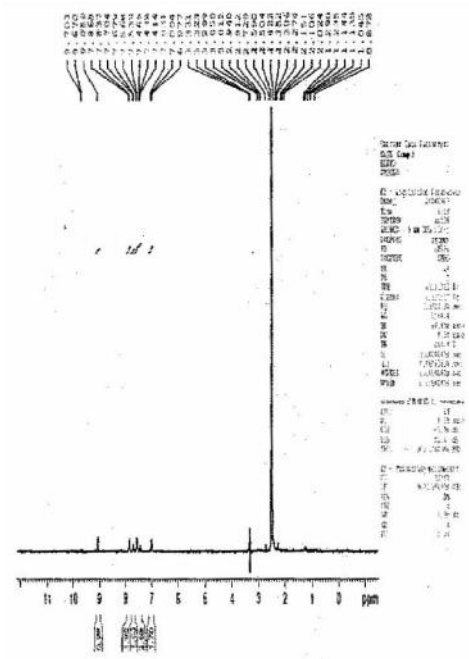


Fig (1) HNMR spectrum of compound(1)

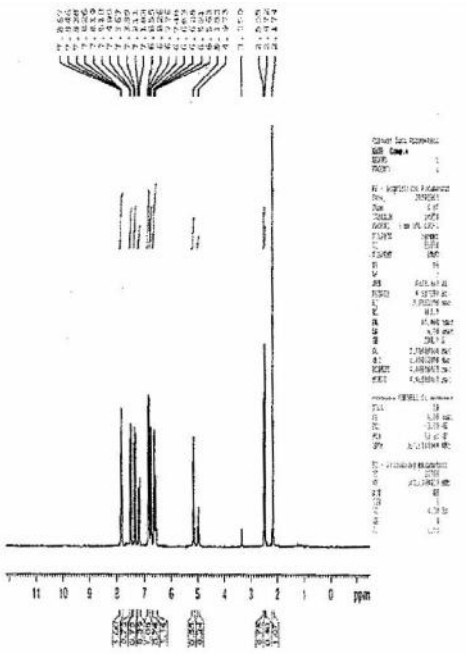


Fig (2) HNMR spectrum of compound(4)

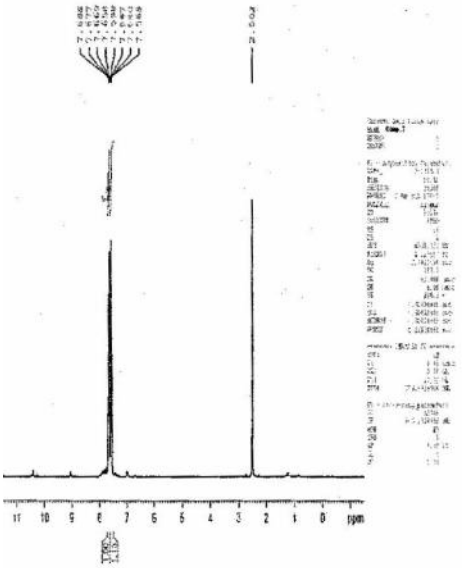


Fig (3) HNMR spectrum of compound(7)

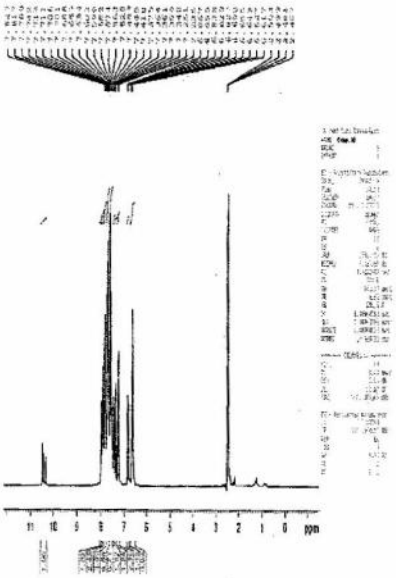


Fig (4) HNMR spectrum of compound(10)

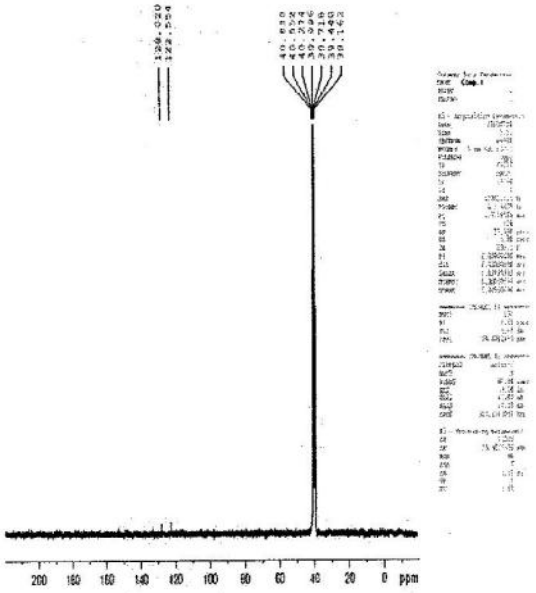


Fig (5) ^{13}CNMR spectrum of compound(1)

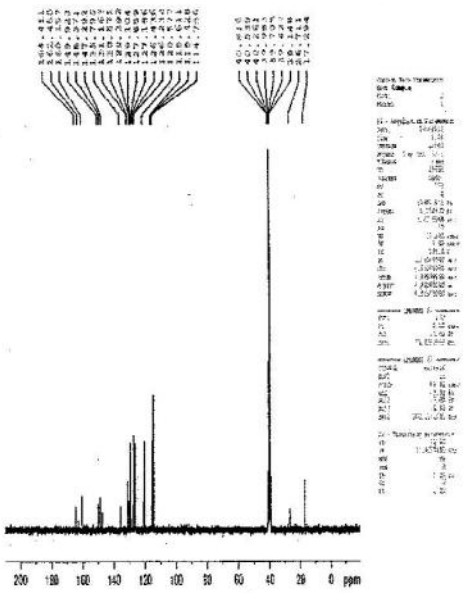


Fig (6) ^{13}CNMR spectrum of compound(4)

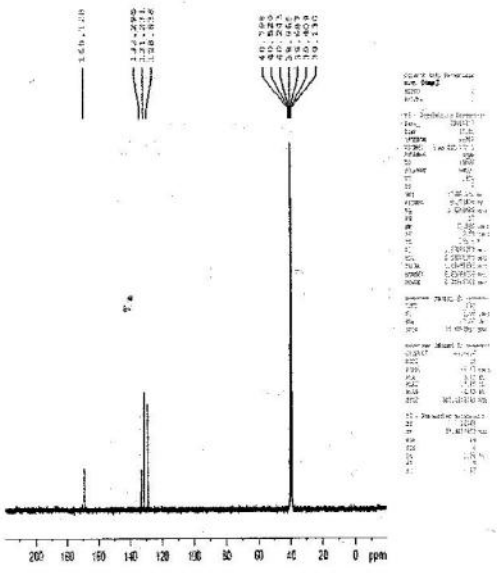


Fig (7) ^{13}CNMR spectrum of compound(7)

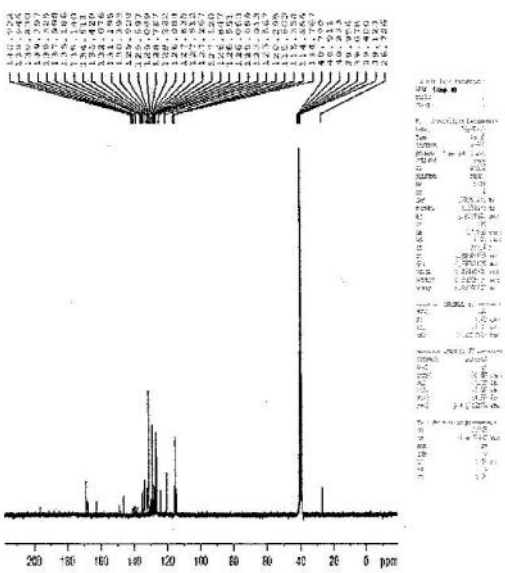


Fig (8) ^{13}CNMR spectrum of compound(10)

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