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Synthesis, Characterization and Biological Investigation of Some New Hydroxy – 1,3,4 – Oxadiazole Derivatives

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ABSTRACT

1,3,4 – Oxadiazole are important compounds because of its versatile biological action such as antimicrobial, antimalarial, anticonvulsant, hypoglycemic, also analgesic, anti – inflammatory and anticancer. In the present study the solid compounds of hydroxy 1,3,4 – oxadiazoles have been synthesized by reaction of acid hydrazides with CS_2 in the presence of KOH. All the prepared compounds are characterized by using elemental analysis (C, H, N and S) and spectroscopic IR, H^1 – NMR and studied the biological activity on four antibacterial (gram +Ve and gram –Ve) and three antifungal organisms.

Keywords: *Oxadiazole, 1,3,4 – oxadiazole, 5 – hydroxyl phenyl oxadiazole and biological activity of oxadiazole.*

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تحضير، تشخيص تقييم بايولوجية لبعض مشتقات هايدروكسي – 4،3،1 – أوكسادايازول الجديدة

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الملخص

تعتبر مركبات 4،3،1 – أوكسادايازول من المركبات المهمة بسبب فعاليتها البايولوجية مثل مضاد للبكتريا، مضاد للملاريا، مضاد للاختلاج وايضا يستخدم كمسكنات ومضاد للسرطان. حضرت المركبات الصلبة من هايدروكسي 4،3،1 – أوكسادايازول في هذه الدراسة من تفاعل هيدرازيدات الحامض مع ثنائي كبريتيد الكاربون بوجود هيدروكسيد البوتاسيوم. تم تشخيص جميع المركبات المحضرة بواسطة التحليل الدقيق للعناصر والتحليل الطيفي (IR, H^1 – NMR) مع دراسة الفعالية البايولوجية لاربع مضادات بكتريا (غرام موجب وغرام سالب) وثلاثة بكتريا تعفن.

الكلمات الدالة: أوكسادايازول، 4،3،1 – أوكسادايازول، أوكسادايازول، 5 – هايدروكسي أوكسادايازول والفعالية البايولوجية للاوكسادايازول .

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1. INTRODUCTION

1, 3, 4 – Oxadiazole is a heterocyclic compound with oxygen atom at (1) and two nitrogen atoms at (3) and (4) position. They have been known for about (80) years, it is only in the last decade, the investigations in this field have been intensified. This is because of large number of applications of 1, 3, 4 – Oxadiazoles in the most diverse areas [1].

It also contains broad range of therapeutic oxadiazoles belong to an important group of heterocyclic compoumds having (–N=C – O–) linkage [1]. There are four know isomers of oxadiazole: 1, 2, 4 – oxadiazole (1), 1, 3, 4 – oxadiazole (2), 1, 2, 3 – oxadiazole (3) and 1, 2, 5 – oxadiazole (4). However 1, 3, 4 – oxadiazole and 1, 2, 4 – oxadiazole are better known and more widely studied by researchers because of their many important chemical biological properties Fig. (1) [2].

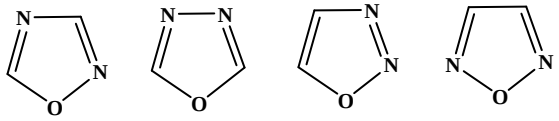

Fig. (1): Structure of isomers of oxadiazole

1, 3, 4 – Oxadiazole (5) is thermally stable aromatic heterocyclic and exists in two partially reduced forms (6); 2, 3 – dihydro - 1, 3, 4 – Oxadiazole and (7) 2, 5 – dihydro - 1, 3, 4 – Oxadiazole depending on position of the double bond. The completely reduced from of 1, 3, 4 – Oxadiazole is designated as 2, 3, 4, 5 – tetrahydro – 1, 3, 4 – Oxadiazole (8) [3] Fig. (2) .

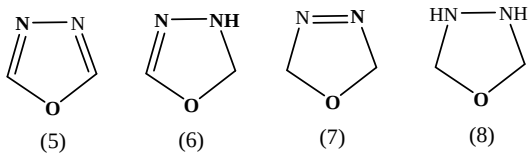

Fig. (2): Structure isomers of 1, 3, 4 – oxadiazole

A number of compounds were prepared depending upon the nature and type of the substituents on the position of benzene ring [4].

We concentrated our attention on 1, 3, 4 – oxadiazole derivatives due to the presence of biological activity of the thiol group and hydroxyl group.

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In this paper, we report the synthesis of hydroxyl derivatives of 1, 3, 4 – oxadiazole and study of biological activity of these compounds [4].

2. Experimental

The Chemicals are used in this study were supplied by different companies such as (Fluka, B.D.H, Merch and GCC) companies. The (C. H. N. and S.) microanalysis and H^1 – NMR recorded in Al – Bayt University / Jordan. The FT – IR spectra are recorded in Al Sulaymania University – Sulaymania / Iraq.

2.1 Synthesis of Propyl Hydroxy Benzoate Derivatives (11-20) [5].

(0.1) mole of hydroxyl benzoic acid derivatives, (1 mole, 50 ml) of propanol (propyl alcohol) and (3) ml of concentrated H_2SO_4 was mixed and the mixture was refluxed for (5) hours, then the excess of propanol distilled off and the residue cooled. (25) ml of crushed ice added to the residue, the precipitate formed, filtered and dried. The physical properties of these hydroxyl esters are listed in Table (1).

Table (1): Physical and analytical data of the esters (11 – 20)

Comp. No.	Compounds Name	Yeild %	M. p. (^{o} C)	M. Wt g/mole	(C.H.N) (cal/found)/ %	
					C	H
11	Propyl Benzoate	80	229 – 230	164.2	73.15 / 73.10	7.37 / 7.11
12	Propyl 3-Hydroxybenzoat	75	200 – 202	180.2	66.65 / 66.50	6.71 / 6.32
13	Propyl 2,3-Dihydroxybenzoate	60	102 – 104	196.2	61.22 / 60.88	6.16 / 5.89
14	Propyl 2,6-Dihydroxybenzoate	80	96 – 97	=	61.22 / 61.05	6.16 / 6.12
15	Propyl 2,4-Dihydroxybenzoate	85	187 – 189	=	61.22 / 60.88	6.16 / 6.25

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16	Propyl 3,5-Dihydroxybenzoate	75	190 – 192	=	61.22 / 61.30	6.16 / 6.25
17	Propyl 2,4,6-Trihydroxybenzoate	60	178 – 180	212.2	56.60 / 56.70	5.70 / 6.00
18	Propyl 2,3,5-Trihydroxybenzoate	70	246 – 248	=	56.60 / 55.99	5.70 / 5.85
19	Propyl 3,4,5-Trihydroxybenzoate	76	268 – 271	=	56.60 / 56.35	5.70 / 5.44
20	Propyl 2,3,6-Trihydroxybenzoate	65	256 – 258	=	56.60 / 56.25	5.70 / 5.36

2.2 Synthesis of Hydroxy Acid hydrazide derivatives from Ester (21-30) [5, 6].

(0.01) mole of ester, (0.02 mole, 15 ml) of hydrazine hydrate (80 %) where mixed in vessel and the mixture was refluxed for (5) hours. After refluxing (30) ml of absolute ethanol added and the mixture was refluxed for (2) hours to complete of the reaction on water bath. The excess of ethanol was distilled off and cooled, the precipitate filtered washed with cold methanol, dried and recrystallized with ethanol. The physical and spectral data are listed in Tables (2) and (4).

Table (2): Physical and analytical data of the acid hydrazides (21 – 30)

Comp. No.	Compounds Name	Yeild %	M. p. (^{o} C)	M. Wt g/mole	(C.H.N) (cal/found)/ %		
					C	H	N
21	Benzohydrazide	85	113 – 117	136.15	61.75 61.55	5.92 5.88	20.58 20.60
22	3-Hydroxybenzohydrazide	70	148 – 149	152.15	55.26 54.89	5.30 5.25	18.41 18.33
23	2,3-Dihydroxybenzohydrazide	67	200 – 202	168.15	50.00 50.17	4.80 4.77	16.66 16.65
24	2,6-Dihydroxybenzohydrazide	77	262 – 264	=	50.00 50.17	4.80 4.78	16.66 16.55

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25	2,4-Dihydroxybenzohydrazide	80	248 – 251	=	50.00 50.12	4.80 4.88	16.66 16.71
26	3,5-Dihydroxybenzohydrazide	68	251 – 253	=	50.00 50.16	4.80 4.78	16.66 16.61
27	2,4,6-Trihydroxybenzohydrazide	69	240 – 242	184.15	45.66 45.55	4.38 4.40	15.21 15.19
28	2,3,5-Trihydroxybenzohydrazide	73	263 – 265	=	45.66 45.71	4.38 4.39	15.21 15.32
29	3,4,5-Trihydroxybenzohydrazide	75	275 – 277	=	45.66 45.59	4.38 4.41	15.21 15.25
30	2,3,6-Trihydroxybenzohydrazide	81	268 – 270	=	45.66 45.68	4.38 4.42	15.21 15.30

2.3 Synthesis of Hydroxy 1,3,4 – Oxadiazole Derivatives from Acid Hydrazides (31-40) [7-10].

(0.01) mole of acid hydrazide derivatives mixed with (0.01 mole, 0.6 g) of KOH dissolved in (1) ml of distilled water in (40) ml of absolute ethanol and (0.01 mole, 1 ml) of CS_2 added dropwise with stirring in cold – water bath, after finishing addition of CS_2, the mixture was refluxed for (5) hours. The excess of ethanol distilled and (30) ml of crush ice was added and acidified to (pH = 2) using (1 N) HCl. The precipitate filtered, dried and recrystallized from (ethanol-water). The physical and spectral properties are listed in Tables (3) and (4).

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Table (3): Physical and analytical data of the oxadiazoles (31 – 40)

Comp. No.	Compounds Name	Yeild %	M. p. (^{o} C)	(C.H.N) (cal/found)/ %			
				C	H	N	S
31	5-Phenyl-1,3,4-oxadiazole-2-thiol	78	187 – 188	53.92 53.88	3.39 3.33	15.72 15.67	17.99 17.89
32	3-(5-Mercapto-1,3,4-oxadiazol-2-yl) phenol	75	201 – 202	49.47 49.34	3.11 2.98	14.42 14.33	16.51 16.55
33	3-(5-Mercapto-1,3,4-oxadiazol-2-yl) benzene-1,2-diol	87	223 – 225	45.71 45.55	2.88 2.86	13.33 13.20	15.25 15.21
34	6-(5-Mercapto-1,3,4-oxadiazol-2-yl) benzene-1,5-diol	85	248 – 250	45.71 46.01	2.88 2.79	13.33 13.35	15.25 15.35
35	4-(5-Mercapto-1,3,4-oxadiazol-2-yl) benzene-1,3-diol	80	261 – 263	45.71 45.88	2.88 2.81	13.33 13.42.	15.25 15.33
36	5-(5-Mercapto-1,3,4-oxadiazol-2-yl) benzene-1,3-diol	75	268 – 270	45.71 45.82	2.88 2.79	13.33 13.42	15.25 15.30
37	6-(5-Mercapto-1,3,4-oxadiazol-2-yl) benzene-1,3,5-tiol	83	378 – 279	42.48 42.46	2.67 2.66	12.38 12.22	14.17 14.11
38	5-(5-Mercapto-1,3,4-oxadiazol-2-yl) benzene-1,3,4-triol	65	251 – 254	42.48 42.55	2.67 2.68	12.38 12.40	14.17 14.20
39	5-(5-Mercapto-1,3,4-oxadiazol-2-yl) benzene-1,2,3-triol	74	269 – 271	42.48 42.51	2.67 2.60	12.38 12.42	14.17 14.19
40	6-(5-Mercapto-1,3,4-oxadiazol-2-yl) benzene-1,4,5-triol	78	258 – 260	42.48 42.53	2.67 2.65	12.38 12.37	14.17 14.25

2.4 Antimicrobial Studies [11-14].

The antimicrobial activity of the synthesized compounds was carried out by cup – plate method using DMSO as a solvent (this solvent not affect to the studied bacteria's or using the solvent without the compound to calibration). In vitro antimicrobial activity was carried out against (24) hour old cultures of four bacteria, namely *E.coli*, *Ps a eruginosa*, *Staphylococcus* and

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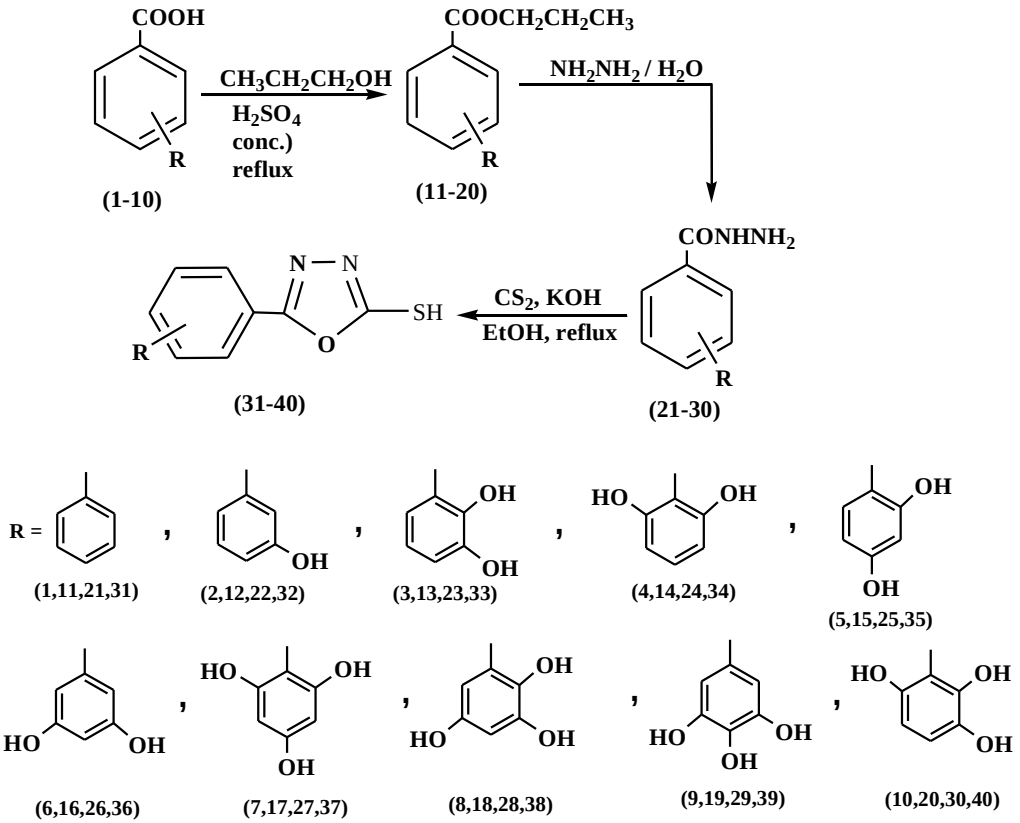
Bacillus Megaterium and (48) hour old culture of three fungi namely *Penicillium*, *Expansum*, *Nigraspora Sp.* And *Trichlothesum Sp.*

3. Results and discussion

Our works of the prepared compounds are illustrated in the following scheme:



Scheme (1)

3.1 Characterization of the Compounds.

The synthesized compounds were subjected to elemental analysis (C, H, N and S) and it present in Tables (1), (2) and (3). They are insoluble in water and soluble in DMSO. The physic - chemical and spectral data of the compounds are proposed to structure of them.

3.2 Infra-Red Spectroscopy (IR) .

IR spectroscopy was used for the characterization of the prepared compounds using KBr compressed disc.

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3.2.1 Infra-Red Spectroscopy of the Acid hydrazide derivatives (17-24).

The most characteristic bands in the IR spectra of the acid hydrazides are the appearance of the stretching vibration of the carbonyl group (amide) in the lower frequency (1680 – 1600) cm^{-1} than the carbonyl groups of the esters that prepared from it at the higher frequency (1750 – 1680) cm^{-1}, another important strong peak is for (-NH) group at (3600 – 3000) cm^{-1} that overlapped with (-OH) phenolic group.

3.2.2 Infra-Red Spectroscopy of the Oxadiazoles (25-32).

The disappearance of the carbonyl group of the amide peak at (1700 – 1650) cm^{-1} and appearance a new peak at (1620 – 1600) cm^{-1} for the (C=N) group with (1390 – 1350) cm^{-1} for the (C=S) group and at (790 – 740) cm^{-1} for (C – S) and for (C – O – C) group at (1070 – 1031) cm^{-1} and (1300 – 1200) cm^{-1} for symmetric and asymmetric absorption.

Table (4): The characteristic infrared bands for the acid hydrazides and oxadiazoles in (cm^{-1})

Co. No.	ν (C=C) ar.	ν (C=N)	δ (N-H) bend.	ν (O-H) ν (N-H)	ν (C-O-C) sym. asym.	ν (S-H)	ν (C=O)	ν (C=S)	δ (O-H) pheno.
21	1587 vs,1520 vs	-	1280 vs	-	-	-	1667 s	-	-
22	1600 vs,1532 vs	-	1279 vs	3320 s,3269 s	-	-	1647 s	-	1367 s
23	1615 vs,1511 vs	-	1268 s	3378 s	-	-	1624 s	-	1355 vs
24	1537 vs,1509 vs	-	1294 vs	3318 vs	-	-	1619 s	-	1326 vs
25	1606 s,1476 s	-	1294 w	3320 s, 3364 s	-	-	1641 s	-	1357 s
26	1622 s, 1515 vs	-	1288 s	3330 s, 3371 s	-	-	1635 s	-	1369 s
27	1630 s, 1520	-	1286 s	3324 s,	-	-	1655	-	1348

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	s			3375 s			s		vs
28	1588 s, 1490 s	-	1278 vs	3340 s, 3387 s	-	-	1644 s	-	1359 s
29	1615s, 1515 s	-	1285 s	3352 s, 3375 s	-	-	1648 s	-	1361 s
30	1610 s, 1520 s	-	1280 s	3348 s, 3379 s	-	-	1655 s	-	1365 s
31	1577 s, 1518 s	1618 vs	1277 s	-	1245 s, 1065 s	2665 w	-	1365 s, 1178 s	-
32	1574 vs,1510 vs	1613 vs	1278 vs	3351 vs	1233 s, 1032 s	2593 w	-	1381 s, 1186 vs	1354 s
33	1533 s, 1520 s	1610 s	1270 s	3345 s, 3380 s	1260 s, 1055 s	2688 w	-	1350 s, 1169 s	1367 s
34	1515 s,1508 s	1614 vs	1268 s	3300 br	1268 s, 1060 s	2590 w	-	1355 vs, 1170 vs	1355 vs
35	1588 s,1508 s	1618 s	1288 vs	3379 w	1288 vs,1071 vs	2780 w	-	1369 vs, 1182 vs	1369 vs
36	1575 s, 1515 vs	1595 s	1279 vs	3378 s	1276 s, 1068 vs	2765 w	-	1370 s, 1180 vs	1355 s
37	1565 s, 1524 s	1611 vs	1280 s	3325 vs	1277 s, 1075 s	2598 w	-	1370 vs, 1178 s	1360 vs
38	1575 s, 1520 s	1598 s	1275 vs	3340 s, 3375 s	1280 s, 1071 s	2678 w	-	1365 s, 1177 vs	1370 s
39	1570 s, 1521 s	1599 s	1278 s	3345 s, 3382 s	1275 s, 1068 s	2665 w	-	1372 s, 1185 s	1375 s
40	1568 s, 1532 s	1615 s	1282 s	3346 s, 3385 s	1278 s, 1070 s	2674 w	-	1369 s, 1180 s	1365 s

ar: aromatic, bend.: bending, sym.: symmetric, asym.: asymmetric, pheno.: phenolic

s: strong, vs: very strong, w: weak, br.: broad

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3.3 Nuclear Magnetic Resonance Spectroscopy (H^1-NMR).

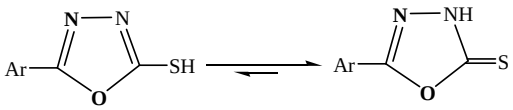
The NMR spectra are recorded using d_6-DMSO as a solvent and TMS as a standard.

3.3.1 NMR Spectroscopy of the Acid Hydrazides (17-24).

NMR spectra of these compounds shows broad singlet peak at (2 – 2.5) ppm for two protons of (NH_2). The (-CO-NH-) protons give singlet at (7.5 – 8) ppm. The aromatic protons appeared in the region (6.8 – 7.9) ppm, whereas the (O – H) protons of the phenolic group appeared as weak singlet at (9.4 – 10.5) ppm.

3.3.2 NMR Spectroscopy of the Oxadiazoles (25-32).

The NMR spectral data of the oxadiazoles showed that the (N –H) and (S – H) protons derived from tautomeric equilibrium resonated at (2.5 – 3.5) ppm as a broad singlet due to the tautomeric forms of the thiol group as shown below [15].



3.4 Antimicrobial Activity.

All the prepared compounds are found to be biologically very active. It is known that hydroxyl and thiol groups act as more powerful and potent bacterial agents. The synthesized compounds were screened for their antimicrobial activity. The zones of inhibition measured around the discs are reported in Table (5) and (6), and comparative analysis is shown in in Figures (3) and (4).

3.4.1 Antibacterial Activity.

The antibacterial activity of the compounds was evaluated against gram positive organisms (*Staphylococcus Aurous)* and (*Becills Megaterium*) and gram negative organisms (*E-coli*) and (*Ps. Aeruginosa*). The zone of inhibition was measured as the parameter of activity.

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Table (5): Antibacterial activity of the oxadiazoles

Co. No.	Zone of inhibition in (mm)			
	Gram +Ve		Gram -Ve	
	Staphylococcus aureus	Becillus megatrium	E - coli	Ps. aeraginosa
31	14	12	18	15
32	18	13	19	14
33	16	17	12	13
34	18	18	14	14
35	19	16	15	15
36	12	17	13	14
37	14	18	14	13
38	15	19	13	16
39	16	18	14	15
40	17	19	16	16

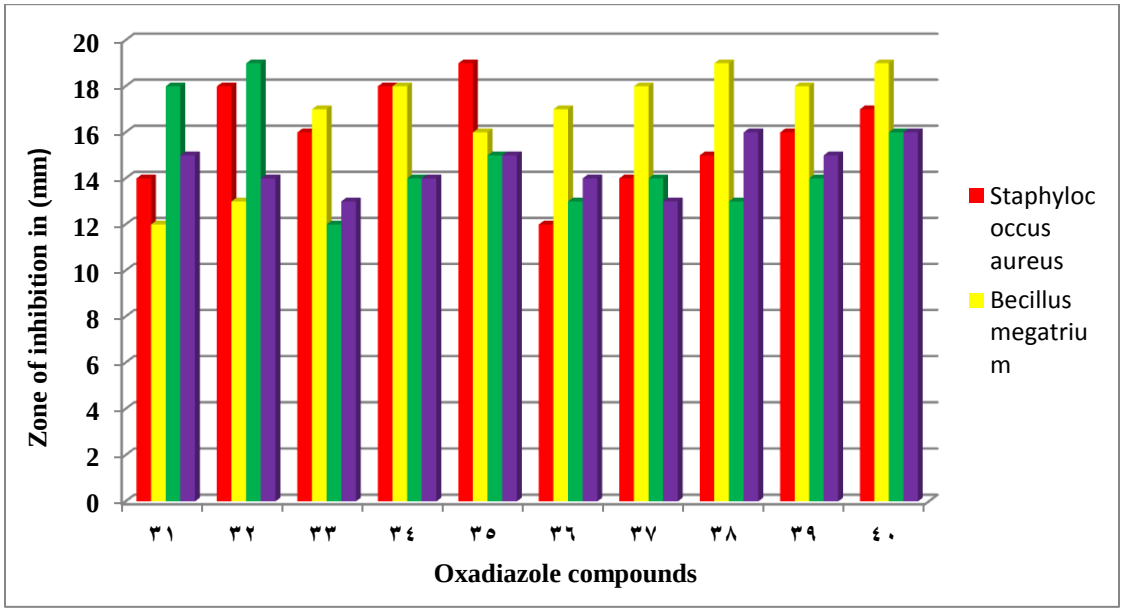

Fig. (3): Comparism of antibacterial activities of the oxadiazoles

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3.4.2 Antifungal Activity.

The examination of antifungal activity of the compounds reveals that the compound (1) moderately toxic against fungi, while the hydroxyl oxadiazoles are more toxic than the compound (1). The activity of the compounds increased when the number of hydroxyl groups increases as shown in Table (5).

Table (6): ***Antifungal activity of the oxadiazoles***

Co. No.	Zone of inhibition at 1000 ppm (%)		
	Penicilium expnsum	Nigras Pora sp.	Trichothesium sp.
31	45	68	63
32	82	80	85
33	70	70	78
34	77	73	79
35	79	75	88
36	80	77	87
37	85	84	89
38	90	89	90
39	85	88	92
40	75	78	82

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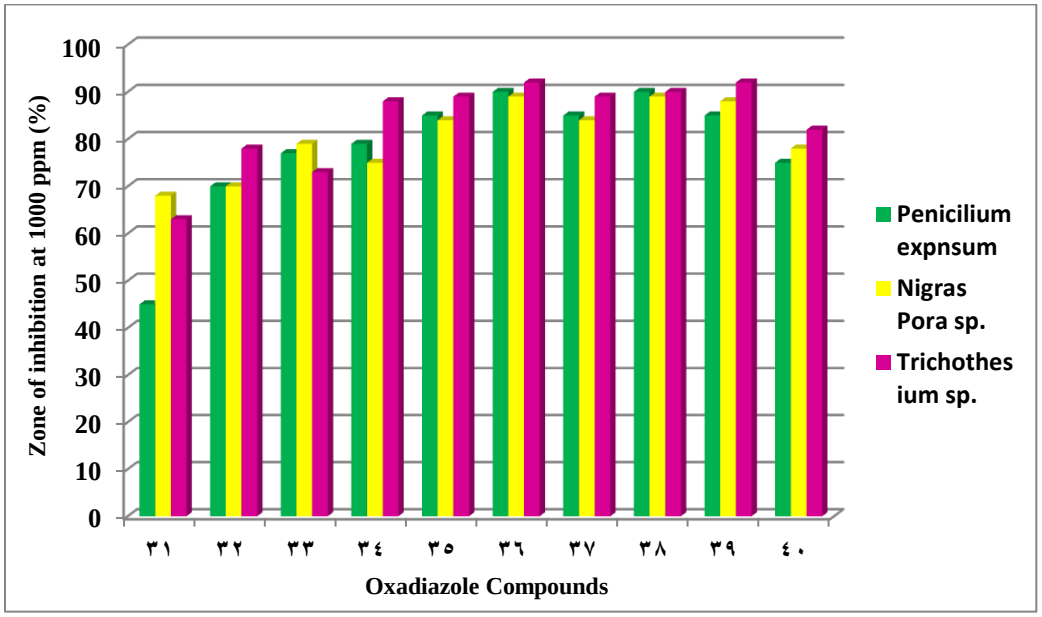

Fig. (4): Comparism of antifungal activities of the oxadiazoles

4. Conclusions

The oxadiazoles were obtained as colored powdered materials and were characterized using physical, spectral (IR and H^1-NMR). The compounds are insoluble in water, ethanol, acetone, ether, chloroform and dichloromethane but soluble in DMF.

From the antimicrobial activity data, it is observed that the oxadiazoles exhibit higher activity than the free hydroxyl oxadiazoles. The increase in antimicrobial activity of the oxadiazoles may be due to the hydroxyl and thiol groups in the compounds.

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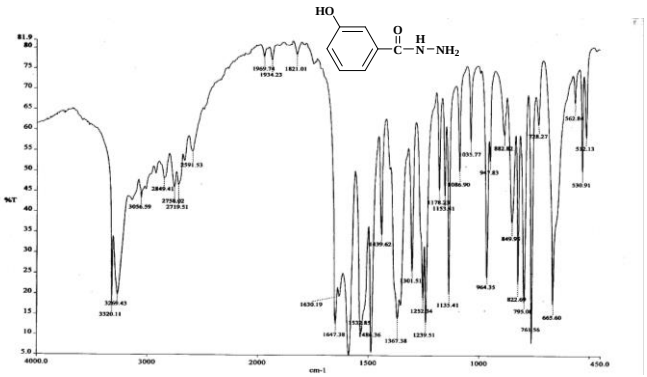

Fig. (5): IR spectra of compound (22)

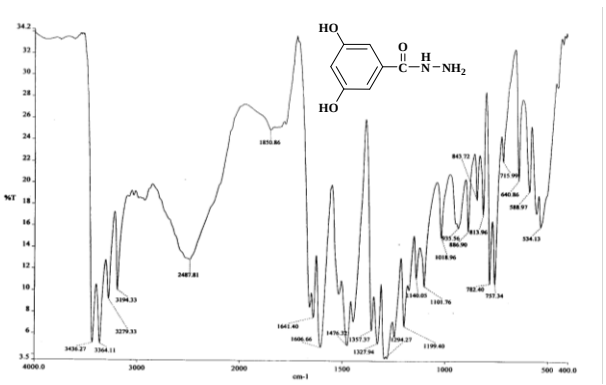

Fig. (6): IR spectra of compound (27)

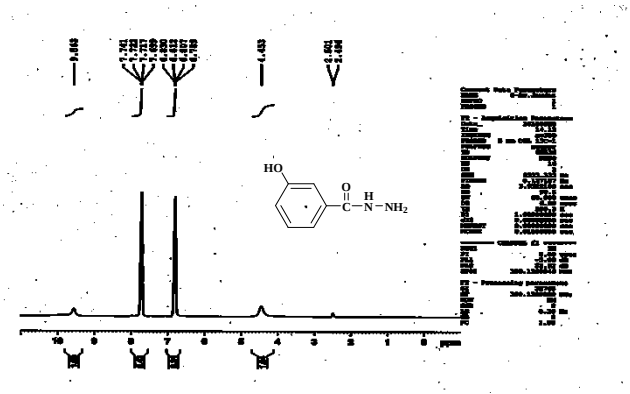

Fig. (7): H^1-NMR spectra of compound (22)

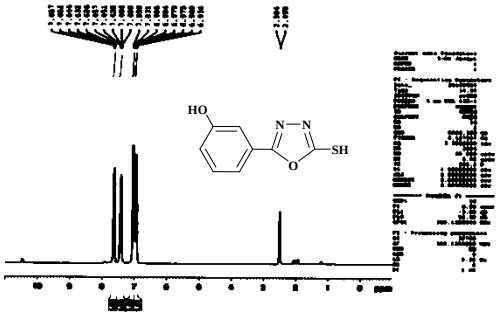

Fig. (8): H^1-NMR spectra of compound (32)

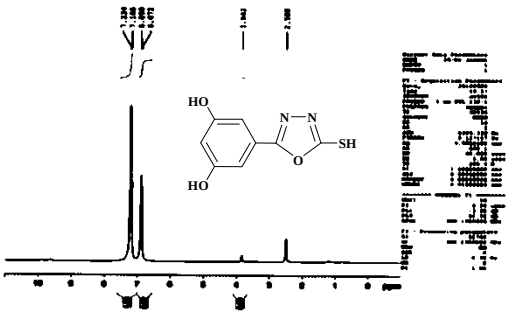

Fig. (9): H^1-NMR spectra of compound (36)

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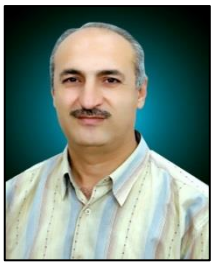

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