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FULL PAPER

Synthesis of new derivatives of 1,3,4-thiadiazole and 1,3,4-oxadiazole on cyclic imides and studying their antioxidant

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The research resulted in the production of new thiadiazole derivatives and oxadiazole derivatives via the reaction of cyclic imides with chloro acetyl chloride at heat condition to give N-acetyl chloro cyclic imides (1,2). Then react these compounds (1,2) with hydrazine hydrate to give N-(aceto hydrazide) cyclic imides (3,4). Then, compounds (3,4) go into three routes: route one was reacting with CS_2 in the presence of (KOH) in ethanol at (24 hrs.) to give (N-[(2-thio-1,3,4-thiadiazolo-5-yl) methylene] cyclic imides) (5,6). Route two was reacting using various aromatic carboxylic acids in the presence of ($POCl_3$) to give (N-[{2-(subs. benzene)-1,3,4-oxadiazolo-5-yl. methylene] cyclic imides) (7-14). Route three was reacting with ammonium thiocyanate in the presence of HCl in ethanol for (5 hrs.), followed cyclization in the presence of concentration (H_2SO_4) to give (N-[(2-amino-1,3,4-thiadiazolo-5-yl) methylene] cyclic imides) (15,16). Prepared compounds have been characterized by FTIR, and some of them by ^{1}H-NMR, melting point, and were studied the effects of the preparing compounds on antioxidant.

KEYWORDS

Cyclic imides; thiadiazole; oxadiazole; antioxidant.

Introduction

Thiadiazole is a unique subclass of five-membered bioactive aromatic hetero-rings containing (1 S atom) and (2 N atoms) [1]. Thiadiazole fraction acts as a "hydrogen binding domain" and "2-electron donor system [2]. There are four isomeric types: 1,2,3-thiadiazole, 1,2,4- thiadiazole and 1,2,5-thiadiazole, 1,3,4-thiadiazole [3]. The therapeutic effect of compounds containing 1,3,4-thiadiazole ring has been well studied for a number of pathological states including antibacterial [4], antimycotic [5], antitubercular [6], anti - Parkinson cancer drug [6], anti-inflammatory [7], and anticonvulsant [8]. There were more studies using 1,3,4-thiadiazole than all other isomers combined [9]. Various 1,3,4-thiadiazole derivatives are of relevance for pharmaceuticals use as well as useful intermediates in the area of organic synthesis [10]. Oxadiazole is an aromatic compound consist of a five-membered ring with 3 hetero atoms (2 nitrogens and 1 oxygen) [11]. One of the most commonly found heterocyclics in medicinal chemistry is 1,3,4-oxadiazoles [12], with broad applications involving anti-bacterial, anticonvulsant, and anti-inflammatory activities [13]. In addition, oxadiazoles 1,2,4-and 1,2,5-are also biologically active, including pesticides [14], insecticides [15], antifungals [16], antibacterial [17], and anti-virals [18]. Anti-oxidant activity is described as the capacity of certain compounds or mixtures to reduce pro-oxidant or reactive species, including free radicals [19].

Experimental

Materials and instrumental

All chemicals used in this study were of the highest purity available and were derived from Fluka, BDH, and Sigma-Aldrich chemicals. The melting point was registered using a Galenkamp capillary melting point apparatus. FT-IR spectra were recorded using KBr disc on Shimadzu FT-IR 8400 Fourier Transform Infrared spectrophotometer in the department of chemistry, college of science, university of Baghdad. Some of the prepared compounds were characterized by 1H-NMR spectra recorded on nuclear magnetic resonance in 400 MHz (Laboratory of Tahran University) with tetramethyl saline as internal standard and dimethyl sulfoxide as a solvent.

Methods

Synthesis of N-acetyl chloro cyclic imides (1,2) [20]

(0.05 mole) potassium imides were suspended in (30 ml) dioxane, then (0.05 mole) chloro acetyl chloride was added to the solution drop by drop at 70-80 C° and refluxed at 6-7 hrs. Then the solution is poured in to crushed ice, and the product form directly, after that filtered then washed by distilled water three times. The products were formed has been dried and recrystallized from EtOH. Table (1) show some of the physical properties and FT-IR spectral data.

Synthesis of N-(aceto hydrazide) cyclic imides(3,4)[21].

(0.01 mole) of N- acetyl chloro cyclic imides were dissolved in (20 ml) absolute ethanol and added 4 drops of Et_3N, then (0.01 mole) of hydrazine hydrate was added drop by drop and refluxed at 4 hrs. The precipitate was collected after the solvent has volatilized, the product was filtered, and washed with diethyl ether, after the precipitate dried, we purified by recrystallized from MeOH. Table (1) show some of the physical properties and FT-IR spectral data.

Synthesis of N-[(2-thio-1,3,4-thiadiazolo-5-yl) methylene] cyclic imides (5,6) [22].

Acid hydrazide compounds (3,4) (0.01 mole) were dissolved in abs. EtOH (30 ml), then KOH (0.01 mole) and carbon disulphide (0.01 mole) respectively were added, and the mixture was stirred for 1 hour. at RT and then refluxed at 80 ºC for 24 hrs. When the solvent was evaporation, the residue dissolved in H_2O (100 ml), acidified with Concentration hydrochloric acid drop by drop was carried out. Red; Yellowish-orange precipitate was collected by filtered, washed with distilled water, and dride. The resulting solid was purified by dissolving in DMSO and reprecipitate from EtOH. Table (1) show some of the physical properties and FT-IR spectral data.

Synthesis of N- [{2-(2-chloro benzen)-1,3,4-oxadiazolo-5-yl} methylene] cyclic imides (7,14) [23]

A mixture of acid hydrazide compounds (3,4) (0.001 mole), different aromatic carboxylic acids (0.001 mole) and $POCl_3$ (3ml) were dissolved in (10 ml) of chloroform, and refluxed for 18 hours. Then the mixture has been cooled and poured in to crushed ice with stirring and neutralized by 10% sodium carbonate solution. The resulting solid was purified by washing three time with distilled water, after that the precipitate was dried, and recrystallized from EtOH. Table (1) show some of the physical properties and FT-IR spectral data.

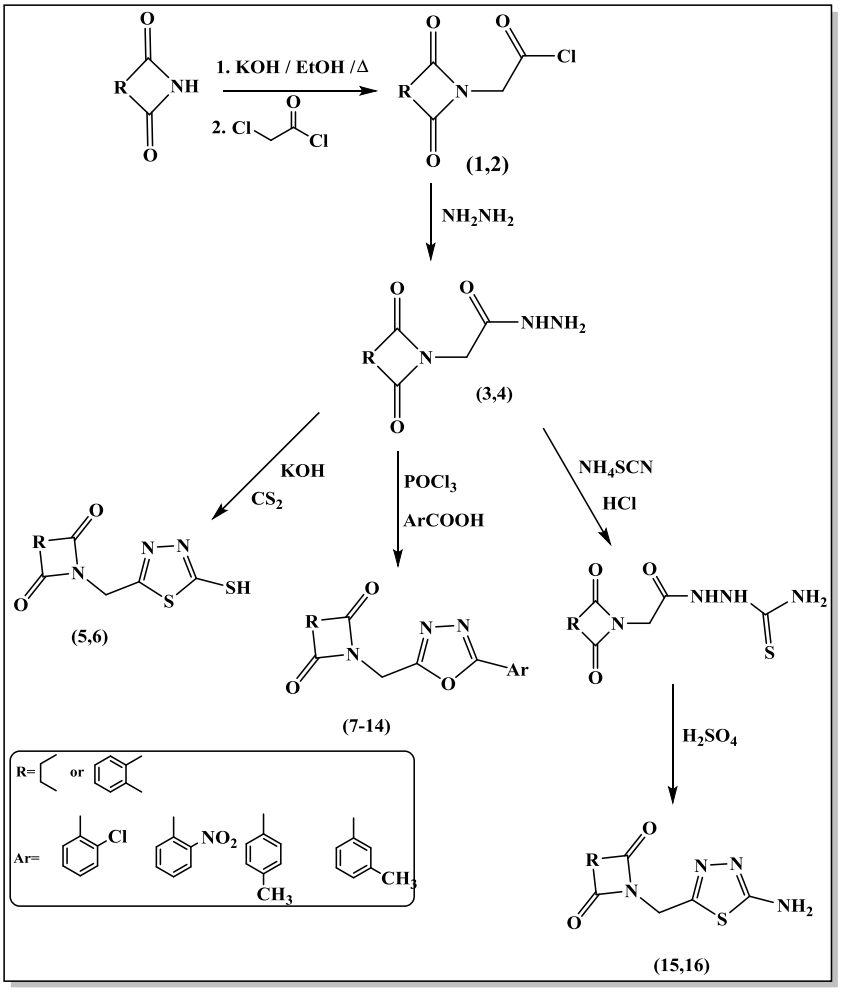
Synthesis of N-[(2-amino-1,3,4-thiadiazolo-5-yl) methylene] cyclic imides (15,16) [24].

Ammonium thiocyanate (0.01 mole) was added in water (5 mL), and hydrochloric acid 1N (3 mL). The reaction mixture was stirred at 100 °C for 45 minute. The produce

isothiocyanic acid intermediate was formed. Acid hydrazide compounds (3,4) (0.01 mole) were added to the reaction mixture and refluxed for (5 hrs.) at 100 °C. Once the reaction is over, the crushed ice was added to reaction mixture and the product was collected by filtration, washed with distilled water and dried. To a mixture of resulting precipitate (0.01 mole) was dissolved in 4 mL of Concentration H_2SO_4. Then the solution was kept at RT for two hours, stirred and poured on crushed ice and neutralized with concentrated aqueous ammonia. The resulting solid has been filtered and purified by washing with water (3 times), dried, and recrystallized from methanol. Table 1 show some of the physical properties and FT-IR spectral data.

Results and discussion

The Present work includes the reaction and synthesis of new derivatives of 1,3,4-thiadiazole and 1,3,4-oxadizole on cyclic imides as shown in Scheme 1.



SCHEME 1

Preparation of N-acetyl chloro cyclic imides (1,2)

These compounds were prepared by reaction of phthalimide or succinimide with chloro acetyl chloride, under heat condition in presence of potassium alcohol, for (6 hrs.). FT-IR spectra of these compounds (1,2) shows the appearance of the absorption bands [2964; 2945, 1805;1803, 1772;1753, 1361;1373, 1269;1294 792;823] cm^{-1} due to υ(C-H) aliphatic, υ(C=O) acid chloride, υ(C=O) imide, υ(C-N), υ(C-O), and υ(C-Cl) respectively, these bands and others absorption bands are shown in Table 1.

Preparation of N-(acetyl hydrazide) cyclic imides (3,4)

N-acetyl chloro cyclic imides react with hydrazine hydrate and tri-ethyl amine as a catalyst to prepare compounds (3,4). FTIR spectra of compounds (3,4) show appearance of the absorption bands [3319;3313, 3222;3257, 1750, 1693, 1440-1425, 1336-1301] cm^{-1} due to $\nu(NH_2)$, $\nu(NH)$, $\nu(C=O)$ imide, $\nu(C=O)$ amide, $\nu(N-N)$ and $\nu(C-N)$ respectively. These and other bands shown in Table 1.

Preparation of N-[(2-thio-1,3,4-thiadiazolo-5-yl) methylene] cyclic imides (5,6)

Compounds (5,6) were prepared by the reaction of compounds (3,4) with CS_2 in the presence of potassium hydroxide in EtOH for (24 hrs.). FT-IR spectra of compounds (5,6) show the appearance of the absorption bands [2972;2932, 2540;2534, 1749; 1752, 1641; 1640, 1448; 1427] cm^{-1} due to υ(C-H) aliphatic, υ(S-H), υ(C=O) imide, υ(C=N) and υ(N-N), respectively. These and other bands are listed in Table (1). Also, disappearance of absorption bands [3319; 3313, 3222;3257,1693;1663.] cm^{-1} due to $\upsilon(NH_2)$, υ(N-H), υ(C=O) amide, respectively, this enhances the success of the ring preparation. ^{1}HNMR spectrum of compound (5) is shown in Table 2.

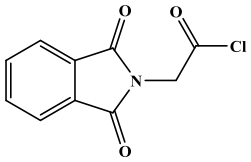
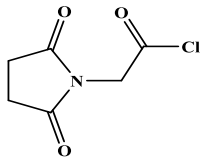
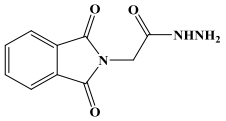
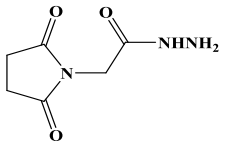
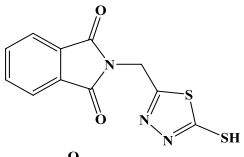
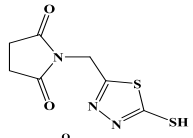
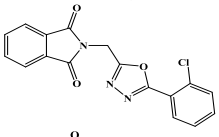
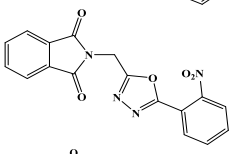
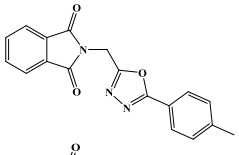
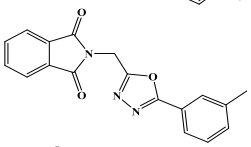
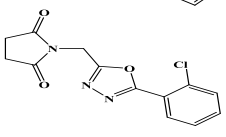
Preparation of N- [{2-(2 or 3 or 4 subs. benzene)-1,3,4-oxadiazolo-5-yl} methylene] cyclic imides (7-14)

Compounds (7-14) were prepared by reaction of compounds (3,4) with different aromatic carboxylic acids in the presence of ($POCl_3$). FT-IR spectral of compounds (7-14) shows the appearance of the absorption bands at [3014-3004, 2983-2887, 1755-1750, 1662-1656, 1602-1600, 1261-1248] cm^{-1} due to υ(C-H) aromatic, υ(C-H) aliphatic υ(C=O) imide υ(C=N), υ(C=C) arom., and υ(C-O), respectively. These absorption bands and others are listed in Table 1. Also, the disappearance of absorption bands [3319; 3313, 3222;2257, 1693;1663.] cm^{-1} due to $\upsilon(NH_2)$, υ(N-H), υ(C=O) amide, respectively, disappearance of these bands enhances the success of the ring preparation. ^{1}HNMR spectrum of compound (9) is shown in Table 2.

Preparation of N-[(2-amino-1,3,4-thiadiazolo-5-yl) methylene] cyclic imides (15,16)

Compounds (15,16) were prepared by reaction of compounds (3,4) with ammonium thiocyanate in the presence of HCl in ethanol for (5 hrs.), followed cyclization in the presence of Concentration H2SO4. FT-IR spectra of compounds (15,16) shows the appearance of the absorption bands [3358;3341, 2900;2923, 1774;1772, 1660,] cm-1 due to υ(NH2), υ(C-H) aliphatic υ(C=O) imide, and υ(C=N) respectively. These and other bands show in Table (1). Also, the disappearance of absorption bands [3222;3257,1693;1663.] cm-1 due to υ(N-H), υ(C=O) amide respectively, disappearance of these bands enhances the success of the ring preparation. 1HNMR spectrum of compound (15) is shown in Table 2.

TABLE 1 Some of the physical properties and FT-IR spectral data cm-1 of the prepared compounds (1-16)

NO.	Physical properties					Major FT-IR spectral data, υ, cm^{-1}					
	Structure	M.P C°	Yield %	Color	NH_2	1. C-H arom. 2. C-H aliph.	1. C=O imide 2. C=O amide	1. N-N 2. C-N	1. C-O 2. C-S	C=N	Other bands
1		120-124	88	Off white	-	**1.**3047 **2.** 2964 2910	**1.** 1753 **2.** -	**1.** - **2.** 1361	**1.** 1269 **2.** -	-	C=O Acid chloride 1803 C=C 1600 1558 C-Cl 792
2		98-102	61	Off white	-	**1.** - **2.** 2945 2823	**1.** 1772 **2.** -	**1.** - **2.** 1373	**1.** 1294 **2.** -	-	C=O Acid chloride 1805 C-Cl 823
3		270 Dec.	81	White	3319	**1.** 3047 **2.** 2920 2850	**1.** 1750 **2.** 1693	**1.** 1440 **2.** 1336	**1.** 1290 **2.** -	-	C=C arom. 1602 1554 N-H 3222
4		135-138	64	White	3313	**1.** – **2.** 2985 2937	**1.** 1748 **2.** 1663	**1.** 1425 **2.** 1346	**1.** 1267 **2.** -	-	N-H 3257
5		211-216	77	Red	-	**1.** 3058 **2.** 2972 2898	**1.** 1749 **2.** -	**1.** 1448 **2.** 1332	**1.** 1234 **2.** 674	1641	C=C arom. 1600 S-H 2540
6		188-192	61	Yellowish orange	-	**1.** – **2.** 2932 2891	**1.** 1752 **2.** -	**1.** 1427 **2.** 1328	**1.** 1220 **2.** 665	1640	S-H 2534
7		262-266	64	Off white	-	**1.** 3006 **2.** 2980 2896	**1.** 1751 **2.** -	**1.** 1448 **2.** 1375	**1.** 1259 **2.** -	1662	(C=C) arom. 1602 (C-Cl) 790
8		> 300	60	Off white	-	**1.** 3014 **2.** 2983 2898	**1.** 1750 **2.** -	**1.** 1440 **2.** 1350	**1.** 1261 **2.** -	1660	(C=C) arom 1602 (NO_2) Asym 1494 Sym 1375
9		> 300	55	White	-	**1.** 3010 **2.** 2980 2893	**1.** 1750 **2.** -	**1.** 1455 **2.** 1347	**1.** 1252 **2.** -	1660	(C=C) arom 1600
10		> 300	48	Off white	-	**1.** 3004 **2.** 2945 2887	**1.** 1751 **2.** -	**1.** 1449 **2.** 1341	**1.** 1250 **2.** -	1658	(C=C) arom 1600
11		210-214	58	White	-	**1.** 3009 **2.** 2957 2988	**1.** 1753 **2.** -	**1.** 1441 **2.** 1338	**1.** 1255 **2.** -	1656	(C=C) arom 1600 (C-Cl) 754

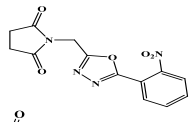
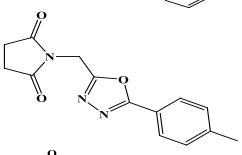
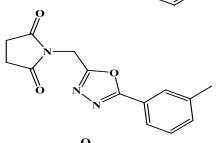
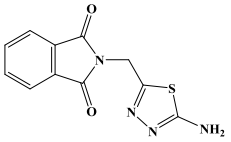
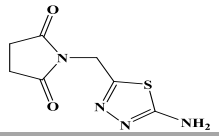
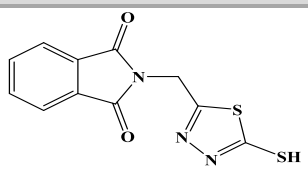
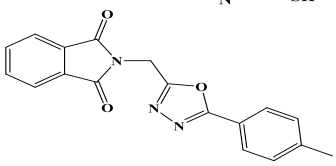
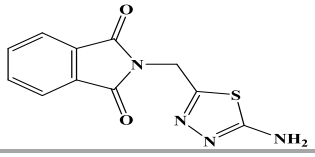
12		265-270	42	Off white	-	**1.** 3012 **2.** 2932 2890	**1.** 1750 **2.** -	**1.** 1447 **2.** 1340	**1.** 1248 **2.**-	1660	(C=C) arom 1601
13		> 300	50	White	-	**1.** 3014 **2.** 2957 2896	**1.** 1755 **2.** -	**1.** 1450 **2.** 1348	**1.** 1251 **2.** -	1660	(C=C) arom 1600 (NO_2) Asym 1501 Sym 1392
14		> 300	45	White	-	**1.** 3007 **2.** 2951 2892	**1.** 1752 **2.** -	**1.** 1443 **2.** 1335	**1.** 1254 **2.**-	1661	(C=C) arom 1600
15		274-278	69	White	Asym 3358 Sym 3283	1. 3020 **2.** 2900	**1.** 1774 **2.** -	**1.** 1438 **2.** 1350	**1.** 1261 **2.** 684	١٦٦٠	(C=C) arom 1602 1557
16		231-236	56	White	Asym 3341 Sym 3266	**1.** – **2.** 2923 2894	**1.** 1772 **2.** -	**1.** 1442 **2.** 1346	**1.** 1253 **2.** 669	1660	-

TABLE 2 ^{1}HNMR spectral data (δ ppm) for some compounds

No. of Comp.	Structure	^{1}HNMR spectral data (δ ppm)
5		4.66 (s,2H, C<u>H</u>$_2$); 7.83 (s, 4H, Ar-<u>H</u>); 13.1 (s, 1H, S<u>H</u>).
9		2.33 (s, 3H, C<u>H</u>$_3$); 4.61 (s,2H, C<u>H</u>$_2$); 6.62-8.09 (dd, 8H, Ar-<u>H</u>).
15		4.83 (s,2H, C<u>H</u>$_2$); 6.85 (s, 2H, N<u>H</u>$_2$); 7.76 (s, 4H, Ar-<u>H</u>).

Antioxidant activity[25]

DPPH Radical Scavenging Activity:

- DPPH (1,1-Diphenyl-2-picryl-hydrazyl): DPPH (4 mg) was dissolved in 100 mL of ethanol, and the solution was kept protected from light by covering the test tubes with aluminum foil.
- Various concentrations of 100, 50, 25, 12.5, 6.25 ppm were prepared from some of prepared compounds. It was prepared by dissolving 1 milligram of the compound and dissolving it with 10 mL of ethanol to prepare 100 ppm, then it was diluted to 50 and 25 ppm…etc.
- Ascorbic acid (vitamin C): Similar concentrations were prepared. as shown in Table 3.

DPPH method

Based on DPPH's stable free radical sweep effect, the antioxidant function of some selective synthesized of some prepared compounds, and a normal (vitamin C) was assessed using the process. In a test tube, 1 mL of the diluted or normal solution (6.25, 12.5, 25, 50, 100 ppm) was applied to 1 mL of DPPH solution. The absorbance of each solution was measured at 517 nm using a

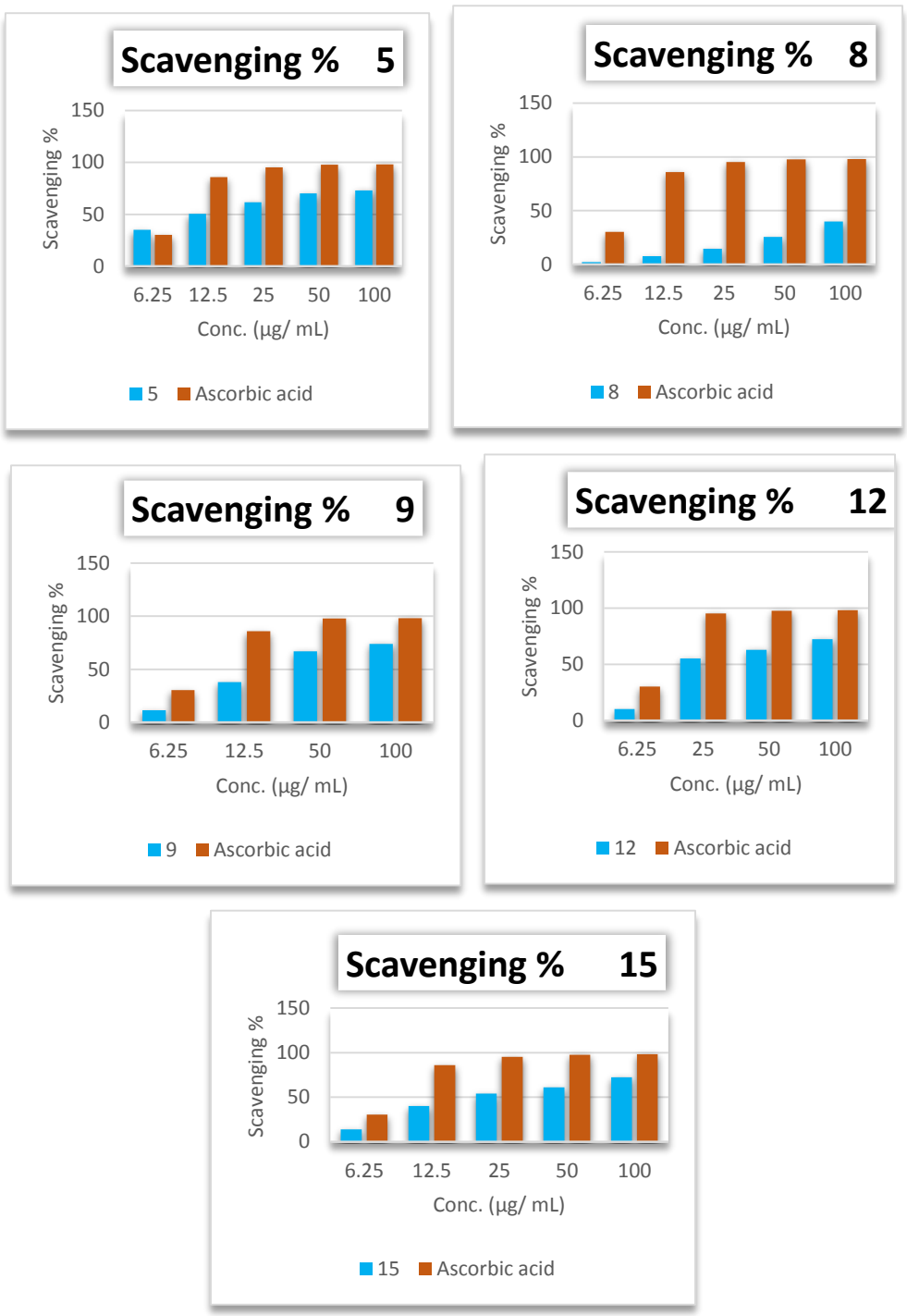
spectrophotometer after 1 hour of incubation at 37 °C. The following equation was used to determine the potential to scavenge DPPH radicals.

I%= (Absorption blank – Absorption sample) / Absorption blank x 100.

Some of the newly prepared compounds showed antioxidant activity against DPPH free radicals and give a good scavenging percentage. So, the compounds that gave antioxidants were selected, more tests were performed, and the (IC_{50}) value was extracted as shown in Table 4.

TABLE 3 % Scavenging for some of prepared compounds

Comp. No.	Conc. (µg/ mL)	Scavenging %
	6.25	35.2
	12.5	50.5
5	25	61.7
	50	70.2
	100	72.9
	6.25	2.1
	12.5	7.7
8	25	14.7
	50	25.6
	100	39.8
	6.25	11.5
	12.5	37.9
9	25	51.2
	50	66.9
	100	73.8
	6.25	10.4
	12.5	31.7
12	25	55.4
	50	62.8
	100	72.5
	6.25	13.7
	12.5	40
15	25	53.9
	50	60.9
	100	72.3
	6.25	30.4
	12.5	85.8
Ascorbic acid	25	95.2
	50	97.7
	100	98.1



These figures show the scavenging's comparison between the prepared compounds and ascorbic acid.

The IC_{50} value of DPPH radical scavenging activity [26]

The IC_{50} value was determined to assess the sample concentration required to inhibit 50% of the radical. The higher the antioxidant activity of compounds, the lower the IC_{50} value. The highest antioxidant activity was found in compound (5), due to NO_2, which is the preferred place of free radical attack [27], and the highest IC_{50} was found in compound (8) as shown in Table 4. Ascorbic acid is a standard with an IC_{50} value of 36.3 ppm.

TABLE 4 IC_{50} value of DPPH radical scavenging activity

Comp. No.	Linear Equation	IC_{50}
5	y = 0.9905x	50.47
8	y = 0.4307x	116.09
9	y = 0.9422x	53.06
12	y = 0.9186x	54.43
15	y = 0.9165x	54.55
Ascorbic acid	y = 1.3767x	36.31

Antioxidant Activity according to Phongpaichit, 2007 [28]

- DPPH assay. IC_{50} > 100 – 250 mg mL^{-1}, weakly active; > 50 – 100 mg mL^{-1}, moderately active; 10 - 50 mg mL^{-1}, strongly active; < 10 mg mL^{-1}, very strongly active.

Conclusion

The prepared compounds were confirmed by using spectroscopic techniques (FT-IR and ^{1}HNMR). The oxidative efficiency of the most compounds (5, 9,12,15) were strong. As opposed to using ascorbic acid as a normal material, one of the performed poorly as antioxidants.

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