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Novel synthesis of 1,3,4 oxadiazole derivatives poly (maleimide-co-methyl acrylate) with some carboxylic acids and their biological activity

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Aromatic polyimides (PIs) are receiving substantial interest because of their brilliant complete properties, multipath synthesis, various processing methods and wide application fields. The current work aimed at the synthesis of a new series of 1,3,4-oxadiazole derivatives on poly (maleimid-co-methyl acylate). In first step poly (maleimid-co-MA) (1) was prepared using gebrail reaction to give poly (potassium maleimid-co-methyl acrylate) (2), then it reacted with different alkyl halide to obtain compounds (3-5) of free radical copolymerization. In the second step it was treated with pure hydrazine (99%) to give compounds (6-8). The final step was the reaction of different carboxylic acid with copoly acid hydrazide in present phosphoryl chloride to give compounds (9-23) as shown in Scheme (1). All prepared compounds were characterized by Softening points (FT-IR), and some of them by HNMR and Thermal gravimetric analysis **(**TGA)). Further, their Biological application was investigated. Some of their physical characteristics are listed in Tables 1 and 2.

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KEYWORDS

Addition polymerization; alternating copolymer; free radical polymerization; 1,3,4-oxadiazole; biological activity.

Introduction

Recently, the improvement of high-performance PI substances has found a significant status [1]. Aromatic polyimides (PIs) are receiving remarkable attention because of their brilliant complete properties, multi-path synthesis, various processing methods and wide application fields [2], the important products protecting PI membranes [3–6], composite materials [7–9], laminated resin [10], coatings, adhesives [11], separation membranes [12], fibers [13], photosensitive substances [14] and liquid crystal alignment layers [15].

Oxadiazoles are heterocyclic five-atom compounds containing one oxygen and atom two nitrogen atoms in their structure. Different types of oxadiazole isomers can be distinguished, occurring in the structure of many drugs [16]. The most promising formulations are 1,3,4-oxadiazole derivatives. The first derivatives of 1,3,4-oxadiazole were prepared at the end of 19th century. The strategies for acquiring the new constructions have been multidirectional, which include the interactions of phosgene and the fantastic hydrazides, and the thermal cycle of the cyclization of 1,2-diacylhydrazines or 1-acylsemicarbazides with the help of drying action [17]. Scientists are currently using various methods to prepare 1,3,4-oxadiazole derivatives, some of which are improved prior

methods, for example, reactions carbon disulfide with cyclooxygenase, cyclohydration, N-acylhydrazones, hydrazide or diacylhydrazines [18]. A massive increase in research on the 1,3,4-oxadiazole ring has been observed during the past two decades [19].

The 1,3,4-oxadiazole ring has demonstrated a wide range of pharmaceutical functions. According to the literature, for many years scientists around the world have been making new compounds with the basic 1,3,4-oxadiazole, which have confirmed an enormous spectrum of biological activity, including anti-inflammatory [20], antidepressant [21] analgesic [22], antidiabetic [24] and anticancer [24] effects. The unusual antimicrobial activity of compounds containing 1,3,4-oxadiazole ring in its structure has been also reported. Scientists have conducted research for an antifungal [25], antibacterial [26], and an antiviral [27] agent carrying a 1,3,4-oxadiazole scaffold.

Experimental

Materials

All chemical compounds used in this study were of the easiest purity accessible and derived from Fluka, BDH, and Sigma Aldrich chemical compounds. Softening points were determined on thermal microscope reichert thermover 160 (University of Baghdad college of science). FTIR spectra were recorded utilizing KBr disc on Shimadzu FTIR 8400 Fourier Transform Infrared spectrophotometer in the Department of Chemistry, College of Science, University of Baghdad. Some of the organized compounds were characterized through H-NMR spectra recorded on NMR in 400 MHz (Laboratory of Isfahan University) with tetramethyl saline as internal standard and DMSO as a solvent. Thermal analysis was performed using thermal analysis system consisting of TGA50 000801, which was carried out in the Laboratory of Isfahan University.

Synthesis of poly(maleimide-co-methylacrylate) (1) [28]

Copolymerization of maleimid (5 mmol) with methyl acrylate (5 mmol) was carried out in DMSO (50 mL) using AIBN (25 mg) as free radical initiator in bath water for 2h under nitrogen atmosphere. The copolymer MIMA was isolated by precipitation to methanol. The precipitated copolymer was washed with methanol several. The product used was purified by using dissolving in DMSO and reprecipitation from methanol. The physical properties are listed in Table 1.

Synthesis of poly (potassium maleimid-co-methyl acrylate) (2) [29]

Poly (maleimide-co-methylacrylate) (0.05 mole) was dissolved in absolute ethanol (30 mL) in presence of potassium hydroxide at 70-80 °C and refluxed for 6-8 hours. Next, the solution was allowed to dry. The products formed were purified by dissolving in DMF and rerecipitating with acetone. The physical properties are listed in Table 1.

Synthesis of poly (N-subs. maleimid-co-methyl acrylate) (3-5) [29]

The poly (potassium maleimid-co-methyl acrylate) (0.01 mole) was suspended in dioxane (30 mL); alkyl halide (0.01 mole) was then added to the solution drop by drop and stirred for 1 hour. The mixture was then left to dry. Next, the product was purified by dissolving in DMF and reprecipitation in ethanol. The physical properties are listed in Table 1.

Synthesis of poly (N-subs. maleimid-co- acryl acid hydrazide) (6-8) [30]

To a solution of poly (N-subs. maleimid-co-methyl acrylate) (1 mmol) in dioxane (30 mL), hydrazine hydrate (99%) (2 mmol) was added, then the resulting mixture was refluxed

for 6hrs. The product was purified by dissolving in ethanol and reprecipitation from acetone. The physical properties are listed in Table 2.

Synthesis of poly (N-subs. maleimid-co- 1,3.4-oxadiazole)

A. From different aromatic carboxylic acids with $POCl_3$ (9-20) [31]

Different aromatic carboxylic acids (1 mmol) and $POCl_3$ (3 mL) were dissolved in chloroform (10 mL) and refluxed for 18 hours. The reaction combination was cooled and poured into beaten ice by stirring and getting neutralized with a solution of sodium carbonate (10%). The resulting solid was washed three times with water. The product was purified through dissolving in DMF and reprecipitated from the water. The physical properties are listed in Table 3.

B. From chloroacetic acid with $POCl_3$ (21-23) [32]

To a stirred solution of phosphoryl chloride (15 mL), acid hydrazide compounds 6-8 (1 mmol) and chloroacetic acid (1mmol) were added at 0 °C, then reaction mixture was refluxed at 80 °C for 4 h. After the completion of the reaction, the reaction mixture was concentrated and poured into ice cold water (100 mL); the solid material was precipitated out which was filtered out and washed with water. The product was purified through dissolving in DMF and re-precipitated from the water. The physical properties are listed in Table 4.

TABLE 1 Some of the physical properties of compounds (1-5) and their FT-IR spectral data (cm^{-1})

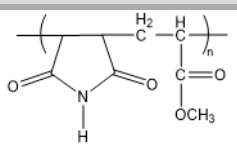
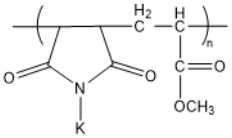
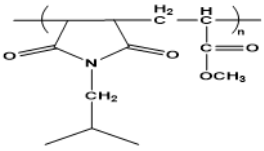
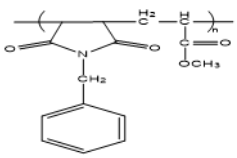
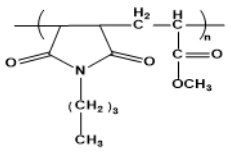
No.	physical properties				FT-IR spectral data (cm-1)					
	Structure	Color	S.P OC	Yield (%)	υ C-H aliph.	1. υ C=O imide 2. υ C=O ester	Υ C-N	υ N-H	υ C-O-C	Other bands
1		Colourless	130-145	82	2954, 2854	1-1724 2-1741	1373	3274	1257	
2		White	200-215	90	2948, 2887	1-1755 2-1739	1373	-	1230	
3		White	140-155	86	2948, 2829	1-1750 2-1693	1373	-	1234	
4		Light yellow	160-175	83	2990, 2836	1-1752 2-1740	1398	-	1220	υ C-H arom. 3044 C=C arom. 1602 1554
5		White	155-170	82.6	2950, 2830	1-1741 2-1730	1350	-	1222	

TABLE 2 Some of the physical properties of compounds (6-8) and their FT-IR spectral data (cm-1)

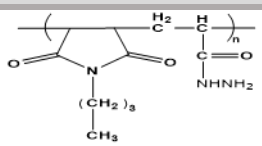
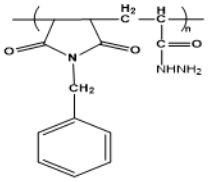
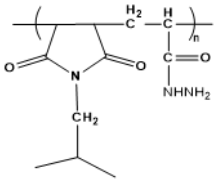
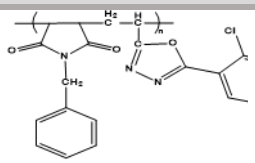
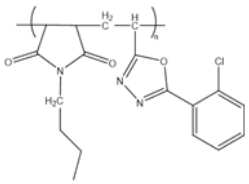
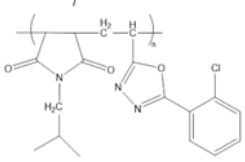
NO.	physical properties				FT-IR spectral data (cm-1)						
	Structure	Color	S.P °C	Yield (%)	C-H aliph.	1. υ C=O imide 2. υ C=O amide	1. υ NH2 2. υ N-H	υ (C-Cl)	υ C-N	υ N-N	Other bands
6		White	125-140	80	2920, 2850	2920, 2850	1-1749 2-1679	1. 3342 2. 3234	1352	1467	
7		Off white	133-147	81	2923 2854	2923 2854	1.1737 2.1668	1. 3325 2. 3220	1371	1473	υ C-H arom. 3039 C=C arom. 1602 1514
8		White	128-142	80	2954, 2854	2954, 2854	1-1730 2-1651	1. 3326 2. 3217	1344	1461	

TABLE 3 Some of the physical properties of compounds (9-20) and their FT-IR spectral data (cm-1)

NO.	physical properties				FT-IR spectral data (cm-1)					
	Structure	Color	S.POC	Yield (%)	1. υ C-H arom. 2. υ C-H aliph	υ C=O imide	υ C=C arom.	1. υ C=N 2. υ C-N	1. υ N-N 2. υ C-O	Other bands
9		Off white	150-165	73	1.3040 2. 2916 2848	1740	1550	1. 1654 2. 1327	1. 1429 2. 1256	υ (C-Cl) 790
10		Off white	148-164	72	1.3044 2. 2928 2856	1730	1545	1. 1658 2. 1320	1. 1425 2. 1259	υ (C-Cl) 795
11		Light yellow	130-145	60	1.3050 2. 2920 2850	1735	1559	1. 1645 2. 1319	1. 1428 2. 1257	υ (C-Cl) 788

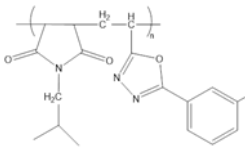
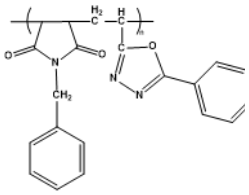
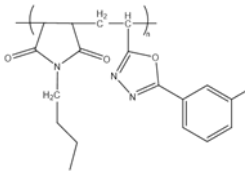
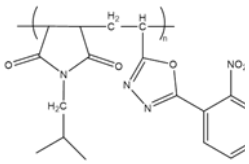
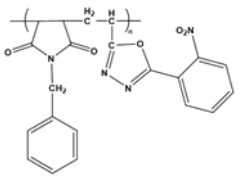
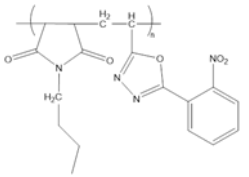
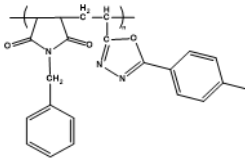
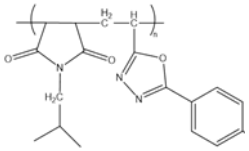
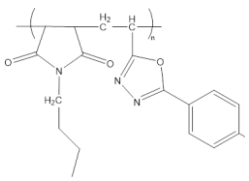
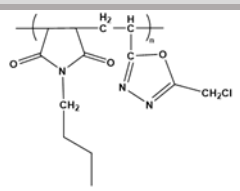
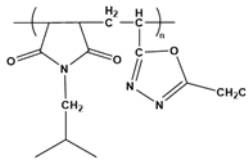
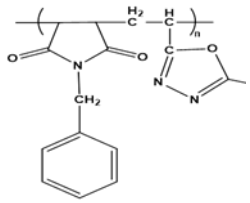
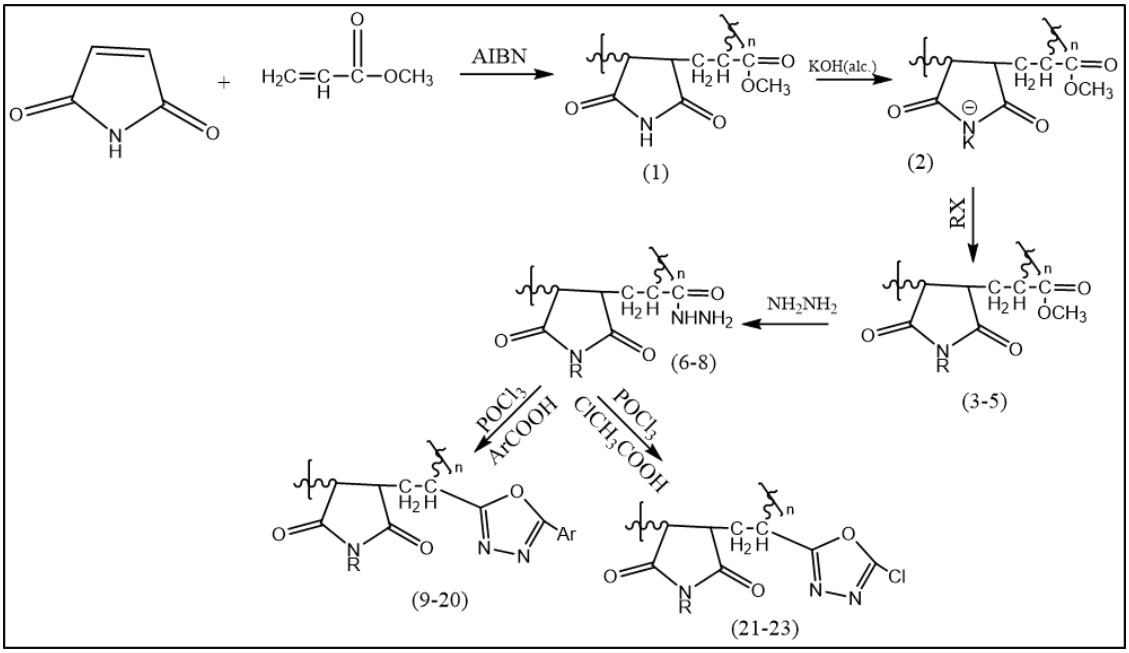
12		Light yellow	160-175	64	1.3047 2. 2929 2856	1744	1559	1. 1659 2. 1328	1. 1429 2. 1259	
13		Light yellow	143-158	62	1.3047 2. 2922 2852	1749	1550	1. 1650 2. 1320	1. 1429 2. 1259	
14		Yellow	155-170	70	1.3040 2.2922 2852	1750	1545	1. 1654 2. 1322	1. 1429 2. 1259	
15		Yellow	154-169	69	1.3045 2.2922 2852	1745	1540	1. 1657 2. 1319	1. 1429 2. 1259	υ (NO2) Asym 1490 Sym 1375
16		Yellow	178-193	72	1.3036 2.2922 2852	1755	1550	1. 1645 2. 1316	1. 1429 2. 1259	υ (NO2) Asym 1494 Sym 1370
17		Light yellow	185-200	68	1.3047 2.2922 2852	1749	1558	1. 1660 2. 1330	1. 1429 2. 1259	υ (NO2) Asym 1484 Sym 1372
18		Light yellow	146-164	67	1.3047 2.2922 2852	1740	1554	1. 1651 2. 1328	1. 1429 2. 1259	
19		Light yellow	150-165	65	1.3047 2.2977 2850	1747	1559	1. 1654 2. 1328	1. 1434 2. 1267	
20		Off white	141-156	75	1.3047 2.2920 2855	1745	1554	1. 1654 2. 1328	1. 1419 2. 1249	

TABLE 4 Some of the physical properties of compounds (21-23) and their FT-IR spectral data (cm^{-1})

NO.	physical properties				FT-IR spectral data (cm-1)					
	Structure	Color	S.POC	Yield (%)	υ C-H aliph.	1. υ C=O imide	υ (C-Cl)	1. υ C=N 2. υ C-N	1. υ N-N 2. υ C-O	Other bands
21		Off white	126-141	76	2928 2853	1740	790	1. 1690 2. 1328	1. 1420 2. 1250	
22		Off white	128-143	78	2926 2859	1749	799	1. 1654 2. 1320	1. 1429 2. 1259	
23		Off white	131-146	77	2922 2852	1753	791	1. 1669 2. 1322	1. 1430 2. 1259	υ C-H arom. 3039 C=C arom. 1602 1514

Results and discussion

This work aimed at the reaction and synthesis of novel derivatives of 1,3.4-oxadiazole as shown in Scheme 1.



SCHEME 1 Synthesis of 1,3,4-oxadiazole

Alternating copolymer preparation of poly(maleimide-Co-methylacrylate)

Maleimide reacted with methylacrylate and AIBN as a catalyst to prepare compounds

(1). The FTIR spectrum of these compounds (1) shows the appearance of the absorption bands [3213, 1724, 1741, 1398,1228] cm^{-1} due to ν (NH), ν (C=O) imide, ν (C=O) ester, ν (C-N) and ν (C-O-C) consecutively. These and other bands are shown in Table 1. ^{1}HNMR spectrum of compound (1) showed signals at δ2.09 ppm (s, 2H, CH-$\underline{CH_2}$); δ 2.96 ppm (t, H, O=C-$\underline{CH}$-CH_2); δ 3.39 ppm (t, H, O=C- $\underline{CH}$-CH_2 imid); δ 3.34 ppm (m, H, O=C-$\underline{CH}$-CH imid): δ 3.59 ppm (s, 3H, O-CH3,); δ 6.67 ppm (s, 1H, NH). Also, there was a signal at δ 2.5 ppm due to the solvent (DMSO).

Preparation of poly (potassium maleimid-co-methyl acrylate)

Poly(maleimide-Co-methylacrylate) reacted with KOH in ethanol as a catalyst to prepare compounds (2). The FTIR spectrum of these compounds (2) shows the appearance of the absorption bands [1755, 1739, 1373,1006] cm^{-1}due to ν (C=O) imide, ν (C=O) ester, ν (C-N) and ν (C-O-C) consecutively. These and other bands are shown in Table 1. The disappearance of the absorption bands at (3213) cm^{-1} due to ν (NH) are also displayed.

Preparation of poly (N-subs. maleimid-co-methyl acrylate)

Poly (potassium maleimid-co-methyl acrylate) reacted with different alkyl halide in dioxane to prepare compounds (3-5). The FTIR spectrum of these compounds (1) shows the appearance of the absorption bands [2990-2818,1750-1741, 1693-1740, 1326-1373,1006-1260] cm^{-1} due to ν (C - H) aliphatic, ν (C=O) imide, ν (C=O) ester, ν (C-N) and ν (C-O-C) consecutively. These and other bands are shown in Table 1: ^{1}HNMR signals for the compound (3) at δ0.96 ppm (d, 6H, CH-$\underline{CH_3}$); δ 3.64 ppm (d, 2H, N- $\underline{CH2}$-CH); δ 2.03 ppm (m, H, CH_2- $\underline{CH}$-CH_3); δ 2.09 ppm (s, 2H, CH-CH_2); δ 2.96 ppm (t, H, O=C-$\underline{CH}$-CH_2); δ 3.39 ppm (t, H, O=C- $\underline{CH}$-CH_2 imid); δ 3.34 ppm (m, H, O=C-$\underline{CH}$-CH imid): δ 3.59 ppm (s, 3H, O-CH_3). Also, there was a signal at δ 2.5 ppm due to the solvent (DMSO).

Preparation of poly (N-subs. maleimid-co-acryl acid hydrazide)

Compounds 3-5 reacted with hydrazine hydrate (99%) in dioxane as a catalyst to prepare compounds (6-8). The FTIR spectrum of these compounds (6-8) shows the appearance of the absorption bands [3303-3326, 3234-3217,1730-1749, 1612-1620, 1398-1328,1434-1467]cm^{-1}due to ν (NH_2), ν (NH), ν (C=O) imide, ν (C=O) amid, ν (C-N) and ν (N-N) consecutively. Also, absorption bands disappear at (1228) cm^{-1} due to ν (C-O-C). These and other bands are shown in Table 2: ^{1}HNMR signals for the compound (7) at δ1.96 ppm (s, 2H, CH-CH_2); δ 2.96 ppm (t, H, O=C-$\underline{CH}$-CH_2); δ 3.39 ppm (t, H, O=C- $\underline{CH}$-CH_2 imid); δ 3.34 ppm (m, H, O=C-$\underline{CH}$-CH imid): δ 3.90 ppm (s, 2H, NH-$\underline{NH_2}$); δ 4.46 ppm (d, 2H, N-$\underline{CH_2}$-Ar); δ 7.21-7.33 ppm (m, 5H, Ar-H); δ 7.50 ppm (s, 1H, O=C-$\underline{NH}$-NH_2). Also, there was a signal at δ 2.5 ppm due to the solvent (DMSO).

Preparation of 1,3.4-oxadiazole

Compounds (6-8) reacted with $POCl_3$ in chloroform as to prepare compounds (9-20). The FTIR spectrum of these compounds (1) shows the appearance of the absorption bands [3040,2916-2848, 1740, 1654,1327,1256] cm^{-1} due to ν (C-H) arom., ν (C - H) aliphatic, ν (C=O) imide, ν (C=N), ν (C-N) and ν (C-O) consecutively. These and other bands are shown in Table 3: ^{1}HNMR signals for the compound (9) at of δ2.96 ppm (s, 2H, CH-CH_2); δ 3.30 ppm (t, H, O-C-$\underline{CH}$-CH_2); δ 3.41 ppm (t, H, O=C- $\underline{CH}$-CH_2 imid); δ3.64 ppm (m, H, O=C-$\underline{CH}$-CH imid); δ 4.46 ppm (d, 2H, N-$\underline{CH_2}$-Ar); δ 7.23-7.93 ppm (m,

9H, Ar-H). Also, there was a signal at δ 2.5 ppm due to the solvent (DMSO).

Compounds (6-8) reacted with $POCl_3$ as a catalyst to prepare compounds (21-23). The FTIR spectrum of these compounds (1) shows the appearance of the absorption bands [3040,2916-2848, 1740, 1654,1327,1256,667] cm^{-1} due to ν (C-H) arom., ν (C - H) aliphatic, ν (C=O) imide, ν (C=N), ν (C-N), ν (C-O) and ν (C-Cl) consecutively. These and other bands are shown in Table 4: ^{1}HNMR signals for the compound (22) at δ 0.93 ppm ppm (d,6H, CH-$\underline{CH_3}$); δ 3.60 ppm (d, 2H, N- $\underline{CH2}$-CH); δ 1.96 ppm (m, H, CH_2- $\underline{CH}$-CH_3); δ 2.59 ppm (s, 2H, CH-$\underline{CH_2}$); δ 3.30 ppm (t, H, O-C-$\underline{CH}$-CH_2); δ 3.34 ppm (t, H, O=C- $\underline{CH}$-CH_2 imid); δ 3.44 ppm (m, H, O=C-$\underline{CH}$-CH imid); δ 4.95 ppm (s, $\underline{CH_2}$-Cl). Also, there was a signal at δ 2.5 ppm due to the solvent (DMSO).

TABLE 5 H NMR spectral data (δ ppm) for some of the prepared compounds

No. of Comp.	1H NMR spectral data (δ ppm)
1	2.09 (s, 2H, CH-CH_2); 2.96(t, H, O=C-$\underline{CH}$-CH_2); 3.39 (t, H, O=C- $\underline{CH}$-CH_2 imid); 3.34 (m, H, O=C-$\underline{CH}$-CH imid): 3.59 (s, 3H, O-CH3,); 6.67 (s, 1H, NH).
3	0.96 (d, 6H, CH-$\underline{CH_3}$); 3.64 (d, 2H, N- $\underline{CH_2}$-CH); 2.03-(m, H, CH_2- $\underline{CH}$-CH_3); 2.09 (s, 2H, CH-CH_2); 2.96 (t, H, O=C-$\underline{CH}$-CH_2); 3.39 (t, H, O=C- $\underline{CH}$-CH_2 imid); 3.34(m, H, O=C-$\underline{CH}$-CH imid): 3.59 (s, 3H, O-CH_3)
7	1.96 (s, 2H, CH-CH_2); 2.96(t, H, O=C-$\underline{CH}$-CH_2); 3.39 (t, H,O=C- $\underline{CH}$-CH_2 imid); 3.34 (m, H, O=C-$\underline{CH}$-CH imid): 3.90 (s, 2H, NH-$\underline{NH_2}$); 4.46 (d, 2H, N-$\underline{CH_2}$-Ar); 7.21-7.33 (m, 5H, Ar-H); 7.50 (s, 1H, O=C-$\underline{NH}$-NH_2).
9	2.96 (s, 2H, CH-CH_2); 3.30 (t,H,O-C-$\underline{CH}$-CH_2);3.41 (t, H,O=C- $\underline{CH}$-CH_2 imid); 3.64 (m, H, O=C-$\underline{CH}$-CH imid); 4.46 (d, 2H, N-$\underline{CH_2}$-Ar); 7.23-7.93 (m, 9H, Ar-H);
12	0.6 (d, 6H, CH-$\underline{CH_3}$); 2.37 (s, 3H, $\underline{CH_3}$-Ar); 3.58 (d, 2H, N- $\underline{CH_2}$-CH); 1.96-(m, H, CH_2- $\underline{CH}$-CH_3); 2.96 (s, 2H, CH-CH_2); 3.30 (t, H, O-C-$\underline{CH}$-CH_2); 3.41 (t, H,O=C- $\underline{CH}$-CH_2 imid); 3.64 (m, H, O=C-$\underline{CH}$-CH imid); 7.19-7.83 (m, 4H, Ar-H)
17	0.86 (t, 3H, CH_2-$\underline{CH_3}$); 1.37 (m, 4H, $\underline{CH_2}$-CH_3); 3.53 (d, 2H, N- $\underline{CH_2}$-CH_2); 1.60 (d, 2H, CH_2- $\underline{CH2}$-CH_2); 2.96 (s, 2H, CH-CH_2); 3.30 (t, H, O-C-$\underline{CH}$-CH_2); 3.41 (t, H, O=C- $\underline{CH}$-CH_2 imid); 3.64 (m, H, O=C-$\underline{CH}$-CH imid); 7.66-8.04 (m, 4H, Ar-H)
18	2.53 (s, 2H, CH-CH_2); 3.30 (t, H, O-C-$\underline{CH}$-CH_2); 3.41 (t, H, O=C- $\underline{CH}$-CH_2 imid); 3.64 (m, H, O=C-$\underline{CH}$-CH imid); 4.61 (d, 2H, N-$\underline{CH_2}$-Ar); 7.19-7.96 (m, 9H, Ar-H); 2.35 (s, 3H, $\underline{CH_3}$-Ar);
22	0.93 (d, 6H, CH-$\underline{CH_3}$); 3.60 (d, 2H,N- $\underline{CH2}$-CH); 1.96 (m, H, CH_2- $\underline{CH}$-CH_3); 2.59 (s, 2H, CH-$\underline{CH_2}$); 3.30 (t, H, O-C-$\underline{CH}$-CH_2); 3.34 (t, H, O=C- $\underline{CH}$-CH_2 imid); 3.44 (m, H, O=C-$\underline{CH}$-CH imid); 4.95 (s, $\underline{CH_2}$-Cl)

Biological activity [32]

Antimicrobial susceptibility tests of some synthesized compounds were performed according to the well diffusion method. A number of synthesized compounds were evaluated on two bacterial strains, one gram positive (Staphylococcus aurous) and one gram negative. (Escherichia coli). The samples were cultured on Muller Hinton agar medium at a temperature of 37 °C for a period of 24 hours, and the results were good for some compounds, as shown in Table 5. Also, one fungal strain like pathogenic fungal (Rhizosporium) was evaluated, where samples were planted on the medium of PDA at a temperature of 28 °C for a period of (3-5) days and some results were good, as shown in Table 6 and Figure 1.

[conc.] = 0.02 g/ml, Control = Solvent = DMSO, Inhibition Zone: (-) no inhibition; (6-10) mm weak; (11-18) mm moderate; (19-30) mm strong, (30-35) mm very strong.

TABLE 6 Anti-microbial for some of the prepared compounds

No. of Comp.	Antibacterial activity test		Antifungal activity test
	Staphylococcus aurous (Gram-positive bacteria)	Escherichia coli (Gram-negative bacteria)	Rhizosporium
Control	-	-	-
1	16	18	10
9	21	20	11
17	23	21	13
18	12	16	20
26	20	22	16
29	22	21	10
Amoxicillin	28	30	
Flucanazole			14

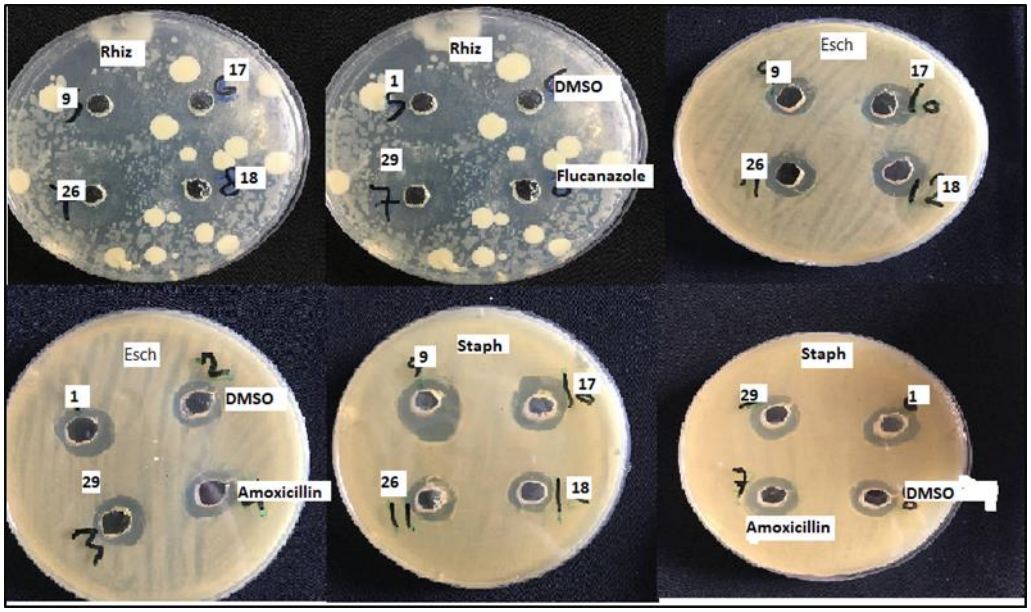


FIGURE 1 Biological activities of some prepared compounds on Rhizosporium, Escherichia coli and Staphylococcus aurous

Antioxidant activity [33]

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 (100, 50, 25, 12.5 and 6.25) ppm were prepared from some of the 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.
- The following equation was used to determine the potential to scavenge DPPH radicals:

I%= (Abs blank – Abs sample) / Abs blank x 100 (Figure 2).

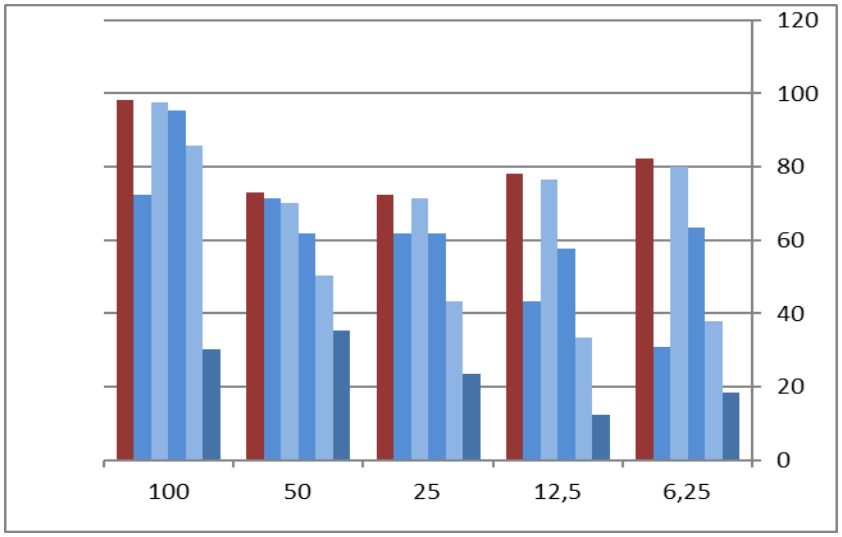


FIGURE 2 The scavenging's comparison between the prepared compounds and ascorbic acid

The IC_{50} value of DPPH radical scavenging activity [34]

The IC_{50} value was determined to assess the sample concentration required to inhibit 50% of the radical. The higher antioxidant activity lowers IC_{50} value of the compound (17) due to NO_2, which is the preferred place of free radical attack. Ascorbic acid is a standard with an IC_{50} value of 36.3 ppm (Table 7).

TABLE 7 IC50 value of DPPH radical scavenging activity

Comp. No.	Linear Equation	IC50
17	y = 1.033x	48
19	y = 0.995x	50
25	y = 0.979x	51.
29	y = 1.01x	49
32	y = 1.02x	46

Thermal gravimetric analysis

Thermal gravimetric evaluation (TGA) measures weight/mass change (loss or gain) and the rate of weight trade as a feature of temperature, time and atmosphere. Measurement is used principally to determine the thermal composition of materials and to predict their thermal stability, revealing that weight loss was below 20 % up to 200 C. The maximum weight loss in 40% occurred between 300 to 600 C. The total weight loss up to 800C is 80.6% (Figure 3).

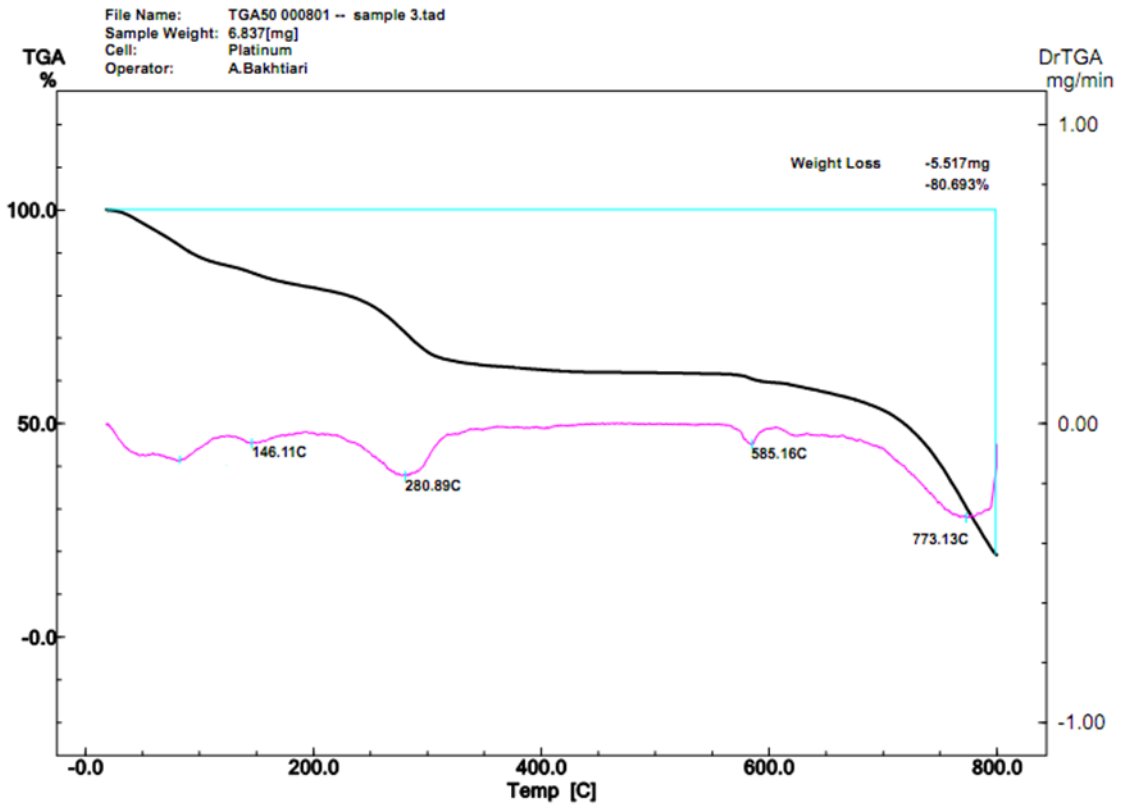


FIGURE 3 Thermal analysis (TG) of compound [18]

Conclusion

The synthesized compounds have been demonstrated with the aid of using spectroscopic methods (FTIR and 1HNMR). Some of the organized compounds gave a top efficiency. The biochemical studies published that the newly synthesized compounds brought about activatory consequences on two types of micro-organism i.e. Staphylococcus aureus, Klebsiella pneumonia, and one type of fungal, i.e. Rhizosporium. Staphylococcus aureus showed reasonable inhibition via the compounds 18 and 26 and excessive inhibition in compounds 9,17 and 29. Klebsiella pneumonia confirmed average inhibition by means of the compounds 9 and 18, excessive inhibition in compounds 17,26 and 29,. Rhizosporium confirmed moderate inhibition in compounds 9,17 and 29 and excessive inhibition in compound 18,26. Based on what achieved, it can be stated that these organized compounds have precise efficacy towards micro-organism and fungi.

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