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Synthesis and Biological Evolution of Novel Substituted 1,2,4-triazine from Sulfanilic Acid

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Abstract

In this paper, a new series of substituted 1, 2,4-triazines was prepared by reacting 4-amino sulfanilic acid with substituted benzaldehyde to form the corresponding Schiff base compounds (1-5), and then reacting two moles of O-methoxy benzaldehyde with hydrazine to form a compound (6) and these compounds reacted with Schiff base (1-5) by applying the Diels-alder reaction in the presence of benzene as a solvent and refluxed it to cyclized into substituted of 1,2,4- triazines derivatives (7-11). Depending on the physical and spectroscopic properties of the prepared compounds that were identified, infrared spectroscopy and nuclear magnetic resonance were used. An evaluation of the biological activity of the prepared compounds was conducted and it gave anti-bacterial results, such as gram-positive and gram-negative when compared with known antibiotics.

*Keywords:*1,2,4-triazine, Sulfanilic Acid

1. INTRODUCTION

Sulfanilic acid is one of the most important types of organic sulfur compounds, which was used as the main nucleus in the preparation of many types of dyes and their derivatives [1,2], especially the mineral formazan dye [3]. Since this substance contains an amino group that can be converted to an (Azo) group [4], in addition to its use in the preparation of benzodiazepine substitutes through reaction of O-phenyl diamine with ketones [5]. The Sulfanilic acid derivatives have been used in the manufacture of materials for use in solar energy applications [6] and some researchers use it as a ligand [7].

Compounds of 1, 2, 4-triazines are prepared for the first time by Bamberger reaction [8] and we can synthesize them in different ways such as from condensation of ethyl oxalamide with diethyl diketosuccinate and hydrolysis to the tricarboxylic acid, followed by thermal decarboxylation and by diels –alder reactions [10] or from oxazolone compounds by reacting it with phenylhydrazine or hydroxylamine hydrochloride [11] . These compounds have a wide interest by researchers because of their biological and pharmacological activity in the fight against AIDS [12,13], in addition to their use as anti-malarial [14], anti-bacterial[15-17], anti-fungal[18], as well as anti-cancer compounds[19]. Moreover, these compounds have industrial applications in the oil industry such as resins, dyes, herbicides, or sulfide removal agents [20].

2. EXPERIMENTAL

2.1. Synthesis of substituted Schiff base(1-5)[21]

A series of Schiff bases (1 - 5) were prepared according to the following procedure: the ethanolic solution of 4-amino sulfanilic acid (0.01 mole in 40 ml absolute ethanol) and (0.01 mol) of substituted benzaldehyde were mixed and refluxed for (6 hrs.), the reaction mixture was evaporated to a small volume and left to cool. The resulting Schiff base precipitated on cooling and then was filtered off, washed with ethanol, and recrystallized from ethanol. Table (1) shows the melting points and physical properties of the compounds.

2.2. Synthesis of bis (O-methoxy benzylidine imine) (6)[22]

(0.02 mole) of O-methoxy benzaldehyde is mixed with (0.01 mole) of (hydrazine hydrate) in the presence of (80%) acetic acid using (30 ml)

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of ethanol, the mixture is stirred for three hours and then cooled. As the solvent evaporates, we get the precipitate with yellow crystals and washed with cold water, then recrystallized using ethanol. The m.p. for this compound (6) was (152-153) with a yield (85%).

Table (1): physical properties and yield of Schiff base compounds (1-5)

No. Comp.	R	m.p.	Yield %	Color
1	2-OH	300 dec.	80	yellow
2	4-Br	< 300	72	white
3	2-methoxy benzaldehyde	300 dec.	80	yellow
4	2-NO_2	>300	72	white
5	3- methoxy benzaldehyde	>300	85	white

2.3. Synthesis of substituted 1,2,4-triazine (7-11) [23]

A series of Schiff bases (1 - 5) were prepared according to the following procedure: the ethanolic solution of 4-amino sulfanilic acid (0.01 mole in 40 ml absolute ethanol) and (0.01 mol) of substituted benzaldehyde were mixed and refluxed for (6 hrs.), the reaction mixture was evaporated to a small volume and left to cool. The resulting Schiff base precipitated on cooling, then was filtered off, washed with ethanol, and recrystallized from ethanol. Table (2) shows the melting points and physical properties of the compounds.

Table (2): physical properties of substituted 1,2,4-triazine compounds (7-11)

No. Comp.	R	m.p.	Yield %	Color
7	2-OH	300 dec.	52	brown
8	4-Br	265-266	53	yellow
9	2-methoxy benzaldehyde	315-316	50	yellow
10	2-NO_2	139-140	51	yellow
11	3- methoxy benzaldehyde	<300	60	yellow

3. RESULTS AND DISCUSSION

The importance of replacing Schiff's bases gave it a great role in the manuscripts of pharmaceutical chemistry, as it is included in many pharmaceutical compounds. Substituted of Schiff base (1-5), these compounds (Scheme1) were synthesized from the reaction of the substituted aldehyde with para-amino acid sulfonate [24], and no catalyst was used, where the acidic proton of sulfonic acid played this role. We note that all the melting points of the products are higher than 300 °C or they disintegrate at the melting point. This is due to the properties possessed by the sulfone group and it corresponds with the literature published online. The data was characterized by main absorption bands of IR spectral data, which gave OH sulfonic acid stretching bands at (2610-2634) cm^{-1}, C=N at (1610-1631) cm^{-1}, and SO2 stretching bands at (1629-1653) cm^{-1}. We note the disappearance of the stretch bands of the amino NH_2 and stretch of aldehyde. Spectra data is shown in Table (3) infrared spectrum and Table (4) nuclear magnetic resonance

Also, Synthesis compound (6) [bis(O-methoxy benzylidine imine)] contains two Imine groups, which are similar in composition to the dienes structure. Hydrazine hydrate (80%) was used to prepare this compound with a ratio of 1 mol from hydrazine to 2 moles of benzaldehyde substitutes, and acetic acid was used as a catalyst to complete the reaction [25-27].

This compound was characterized by the following main absorption bands of IR spectral data C-O-C (1156-1246), (C=N) 1664, C-H aliphatic 2964, C-H Aromatic 3073. The ^{1}H-NMR spectra showed (δ, ppm) : 3.89(s,6H,OCH_3); 7.14,7.16(s,2H,CH=N); 7.04(t,2H, C6 anisole ring); 7.49,7.53(t,4H, (C4,5) anisole ring); 7.98-8.00(d,2H, C2 anisole ring). To prepare the substituted of 1, 2, 4-triazines (7-11) the di-imine compounds were reacted with Schiff bases in a reaction similar to the reactions of Diels-Aldur, where the substitutes for the Schiff bases (1-5) as dinofyl and the diamine compound (6) as diene [23] by reflux was used benzene as a solvent, the mechanism suggestion shown in figure (2) .

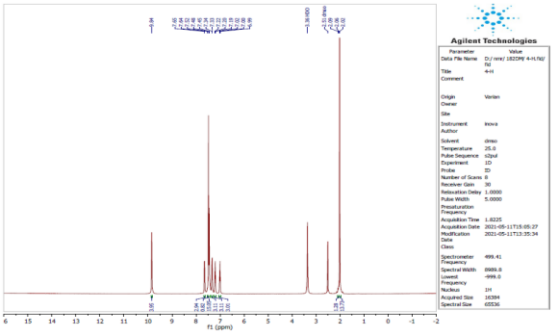


Figure (1): ^{1}HNMR spectra for Compound(4)

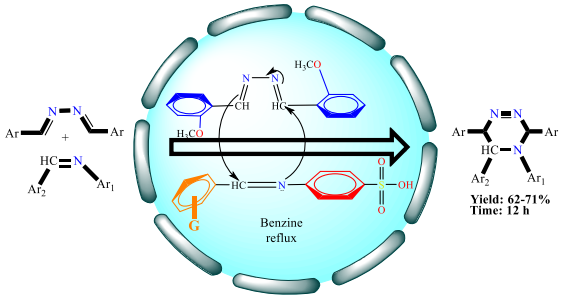


Figure (2): Mechanism of Synthesis 1,2,4-Triazines

Identification of the triazine compounds, spectroscopic methods were used where we observed the O-H stretching bands of sulfonic

acid at the range (2635-2655) cm^{-1}, SO_2 stretching bands at (1613-1657) cm-1 as well as N=N stretching bands at the range (1318-1393) cm-1. And the complete disappearance of the C=N stretching frequencies that were appearing in the initial compounds of the reaction. Spectra data is shown in Table (5) infrared spectrum and Table (6) nuclear magnetic resonance.

Table (3): IR Spectral of Schiff base compounds(1-5)

No. Comp.	R	OH Sulfonic acid	C=N	C-H Aliphatic	C-H aromatic	SO_2	Others
1	2-OH	2610	1610	2942	3030	1653	O-H 3342
2	4-Br	2631	1630	2874	3036	1635	C-Br 829
3	2-methoxy benzaldehyde	2632	1630	2913	3035	1650	O- C 1120
4	2-NO_2	2632	1631	2877	3062	1650	NO_2 sym 1240 Asym 1422
5	3- methoxy benzaldehyde	2634	1613	2963	3063	1629	C-O 1107

Table (4): ^{1}HNMR spectra for Schiff base compounds (1-5)

No. Comp.	^{1}HNMR (δ, ppm)
1	6.97 (s,H,CH=N); 7.31-7.46 (d:d,4H,sulfanilic acid ring); 7.5-7.54 (m,2H,(C3,4)phenol ring); 7.66 (d,H,C5 phenol ring); 7.72 (t,H,C2 phenol ring); 9.05 (s,H,OH phenol ring); 10.27 (s,H,OH sulfonic acid).
2	7.25 (s,H,CH=N); 7.49-7.51 (d:d,4H,sulfanilic acid ring); 7.67-7.95 (d,4H,4-bromobenzene ring); 9.51 (s,H,OH sulfonic acid).
3	3.88(s,3H,OCH3); 6.98 (s,H,CH=N); 7.27-7.96 (d:,4H,sulfanilic acid ring); 7.39-7.44 (d,2H,(C3,4)anisole ring); 7.63 (d,H,C5 anisole ring); 7.78 (t,H,C2 anisole ring); 10.16 (s,H,OH sulfonic acid).
4	6.99 (s,H,CH=N); 7.00-7.22 (d:d,4H,sulfanilic acid ring); 7.33-7.45 (s,2H,(C3,4)nitrobenzene ring); 7.52 (d,H,C5 nitrobenzene ring); 7.65 (t,H,C2 nitrobenzene ring); 9.84 (s,H,OH sulfonic acid).
5	3.36(s,3H,OCH3); 7.24 (s,H,CH=N); 7.26-7.64 (d:,4H,sulfanilic acid ring); 7.67-7.73 (m,2H,(C3,4,5)anisole ring); 7.88 (t,H,C2 anisole ring); 9.6 (s,H,OH sulfonic acid).

Table (5): IR Spectral for triazine compounds (7-11)

Comp. NO.	G	OH Sulfonic acid	C-H Aromatic	C-H aliphatic	C-C aromatic	SO_2	N=N	C-O-C	Other
7	2-OH	2635	3003	2840	1465-1596	1630	1318	1107, 1242	O-H 3400
8	4-Br	2650	3061	2865	1498-1600	1657	1372	1111, 1243	C- Br 829
9	2-OCH_3	2654	3074	2963	1464 - 1598	1614	1319	1108, 1246	C –O 1156
10	2-NO_2	2635	3073	2963	1465-1598	1613	1339	1107, 1246	1546, 1319 $NO_{2\ Sym\ ,\ Asym}$
11	3-OCH_3	2655	3073	2963	1466 - 1598	1613	1393	1156, 1246	

Table (6): ^{1}HNMR for triazine compounds (7-11)

No. Comp.	G	^{1}HNMR (δ, ppm)
7	2-OH	3.84 (d,H,CH C5 triazine ring); 3.89 (d,H,CH C6 triazine ring); 3.94 (s,6H,OCH_3); 7.05-7.49 (t,4H, O-phenol ring); 7.14-7.16 (d,4H,(C3&4) O-anisole ring); 7.24-7.25 (d,2H,(C5) O-anisole ring); 7.71-7.69 (d,2H,(C2) O-anisole ring); 7.98 (s,H,CH sub. O-anisole between N-CH-N); 7.49,7.52(d:d,4H, proton of Sulfonic acid ring); 8.04 (s,H,OH phenol ring); 8.99 (s,H,OH sulfonic acid).
8	4-Br	2.55 (d,H,CH C5 triazine ring); 3.80 (d,H,CH C6 triazine ring); 3.90 (s,6H,OCH_3); 7.16,7.99 (t,4H, P-bromobenzene ring); 7.23-7.24 (d,4H,(C3&4) O-anisole ring); 7.04-7.07 (d,2H,(C5) O-anisole ring); 7.61-7.63 (d,2H,(C2) O-anisole ring); 7.54 (s,H,CH sub. O-anisole between N-CH-N); 6.97,7.69(d:d,4H, proton of Sulfonic acid ring); 8.94 (s,H,OH sulfonic acid)

9	2-OCH_3	3.37-3.86(d,2H,CH,C5and C6 triazine ring); 3.93 (s,9H,OCH3); 6.98-6.95 (t,3H, (C5) O-anisole ring); 7.13-7.16 (d,3H,(C3) O-anisole ring); 7.4 (d,3H,(C4) O-anisole ring); 8.0-8.04 (d,3H,(C2) O-anisole ring); 7.67 (s,H,CH sub. O-anisole between N-CH-N); 7.40-7.41,7.679 (d:d,4H, proton of Sulfonic acid ring); 8.99 (s,H,OH sulfonic acid).
10	2-NO_2	3.81 (d,H,CH, C5 triazine ring); 3.92 (d,H,CH, C6 triazine ring); 3.89 (s,6H,OCH_3); 8.93,7.98,7.73,7.53 (d,4H, O-Nitrobenzene ring); 6.98 (d,2H,(C5) O-anisole ring); 7.07,7.31 (d,4H,(C4&3) O-anisole ring); 7.76 (d,2H,(C2) O-anisole ring); 8.60 (s,H,CH sub. O-anisole between N-CH-N) ; 7.04,7.95 (d:d,4H, proton of Sulfonic acid ring); 9.78 (s,H,OH sulfonic acid
11	3-OCH_3	2.9 (d,H,CH, C5 triazine ring); 3.1 (d,H,CH, C6 triazine ring); 3.89 (s,9H,OCH_3); 7.05,7.50 (t,4H, M-anisole ring); 7.14 (d,4H,(C3&4) O-anisole ring); 7.26-7.27 (d,2H,(C5) O-anisole ring); 7.98 (d,3H,(C2) O-anisole ring); 8.00 (s,H,CH sub. O-anisole between N-CH-N); 7.98 (d,4H, proton of Sulfonic acid ring); 8.95 (s,H,OH sulfonic acid

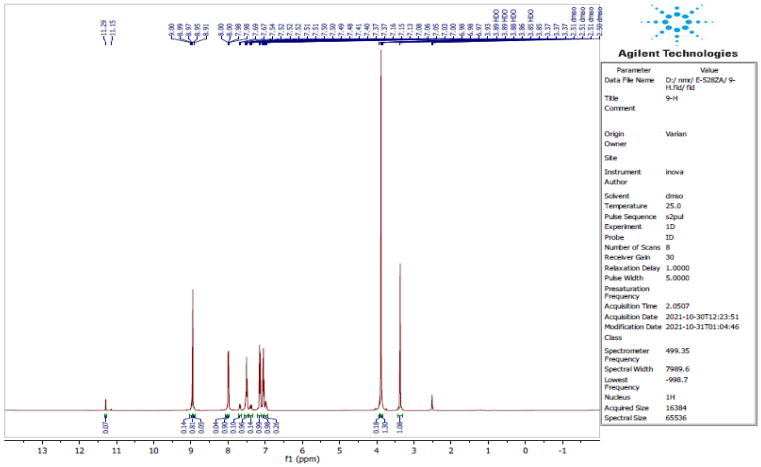
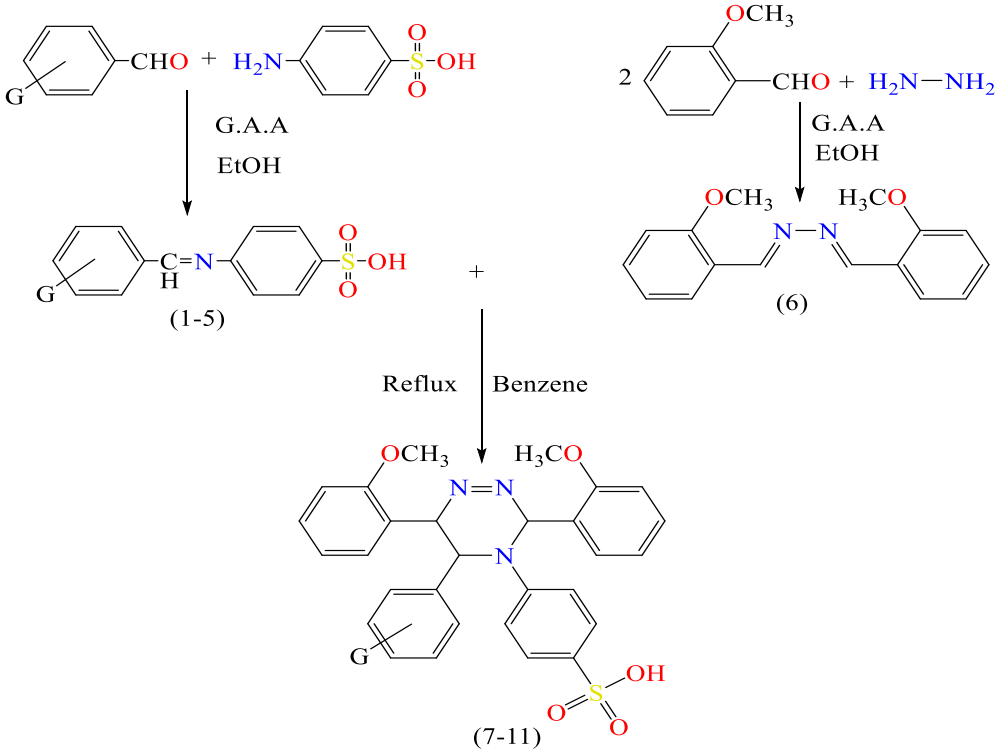


Figure (3): ^{1}HNMR spectra for Compound(9)



G= 2-OH, 4-Br, 2-OCH_3, 2-NO_2, 3-OCH_3

Scheme (2)

4. BIOLOGICAL ACTIVITY

Biological evaluation of the prepared compounds was carried out as they were used as antibacterial for gram-positive and gram-negative bacteria. It is known that the effect of the sulfonic acid group is large, so it gave excellent results if compared with the known antibiotics, where the compounds showed 7-11 higher efficacy than those used [28-30].

Table (7) shows the values obtained by studying the biological activity of the prepared compounds (1-11).

Table (7): biological activity of compounds (1-11)

Compounds No.	Compounds zone of inhibition in mm			
	Staphylococcus aurous	Staphylococcus epidermises	E.coli	Proteus vagaries
Ampicillin	18	18	15	15
Compound 1	16	17	10	9
Compound 2	14	18	16	14
Compound 3	12	13	12	10
Compound 4	15	19	17	17
Compound 5	9	12	13	10
Compound 6	8	14	15	11
Compound 7	21	18	17	18
Compound 8	23	20	20	18
Compound 9	19	21	20	16
Compound 10	26	22	24	21
Compound 11	20	18	16	16

5. CONCLUSIONS

Triazine compounds were created in this research with sulfanilic acid, which had a significant role in the resulting compounds, as it gave them a high activity against anti-bacteria to the high effect of sulfur. These compounds were synthesized in a method similar to the Diels-Alder reactions, where di-imine hydrazone was used with Schiff bases react cyclized to obtain Triazine compounds as the product.

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