ISSN 2664-8075 (Print) & ISSN 2706-5774 (Online)
South Asian Research Journal of Applied Medical Sciences
Abbreviated Key Title: South Asian Res J App Med Sci

| Volume-8 | Issue-5 | Sep-Oct- 2026 | **DOI: https://doi.org/10.36346/sarjams.2026.v08i05.001**

Original Research Article

Synthesis, Characterization and Antibacterian Activity Studies of New Cyclic Compounds 1, 3, 4-Thiadiazole and 1,3-Oxazepine

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Article History
Received: 10.07.2026
Accepted: 04.09.2026
Published: 09.09.2026

Abstract: The synthesis of novel five- and seven-membered heterocycles was conducted using Mefenamic acid as the starting material. Involved treatment of Mefenamic acid with thiosemicarbazide yielded 5-(2-((2,3-dimethyl-phenyl)amino)phenyl)1,3,4-thiadiazol-2-amine [1], and subsequent condensation of this product with 4-N, N-dimethyl amino benzaldehyde, 4-nitro benzaldehyde, 4-bromobenzaldehyde, 4-chlorobenzaldehyde, 4-methoxybenzaldehyde, and 4-hydroxy benzaldehyde gave rise to Schiff bases [2-7]. Other studies involved the treatment of Schiff bases [2-7], with different anhydrides (succinic, maleic, phthalic, and naphthalic) to yield 1,3-oxazepine-4,7-dione rings [8-31]. Focus on the research of the antibacterial properties of some of the synthesized compounds. The well diffusion method was used to determine the in vitro anti-bacterial activity of the *Staphylococcus aurius (G+), Baceillus subtilis (G+), Klebsiella pneum0niae (G-), and E. coli (G-)* bacterial isolates. Some of these compounds demonstrated activity, having been characterized by studies, ^{1}H NMR, ^{13}C NMR, and infrared spectroscopy. Their microbiological activity, however, was not inconspicuous. Indeed, during the antimicrobial activity screening, some of these compounds were found to possess good activity against bacteria (9, 19, 23, 25, and 29).

Keywords: Mefenamic Acid, 1,3,4-Thiadiazole, Schiff Bases, 1,3-Oxazepines-4,7-Dione.

1. INTRODUCTION

Mefenamic acid, a member of the anthracitic acid derivative class of n0n-ster0idal anti-inflammatory drugs (NSAIDs), is used as a potent pain reliever and anti-inflammat0ry agent to treat rheumatoid arthritis, musculoskeletal disorders, and joint cartilage degeneration. It is believed that this effect occurs because it interferes with the biosynthesis of prostaglandins [1, 2].

According to the literature, mefenamic acid undergoes various reactions. These reactions have been utilized to create a range of compounds with heterocyclic therapeutic properties for various applications, including the development of potent anti-cancer medications and agents with antimicrobial, anti-inflammatory, and cytotoxic activities [3, 4].

Because of their wide range of biological functions, 1,3,4-thiadiazoles have emerged as a significant class of heterocyclic compounds and a subject of intense study interest. A five-ring structure with a sulfur atom, a hydrogen-binding domain, and a two-electron donor nitrogen system, thiadiazole exhibits a wide range of biological functions.[5-14] With two heteroatoms, an oxygen atom at position (1) and a nitrogen atom at position (3), 1,3-oxazepines-4,7-dione is a seven-membered ring complex. Oxazepine is characterized by its strong hydrophobicity and poor wettability when it comes into contact with water. 1,3-oxazepines-4,7-dione hydrolyzes very slowly in water solutions, even when alkali is added, and is challenging to degrade or decontaminate at room temperature in real-world situations. 1,3-oxazepines-4,7-dione is a white solid that is stable at high temperatures and has a low [15-21].

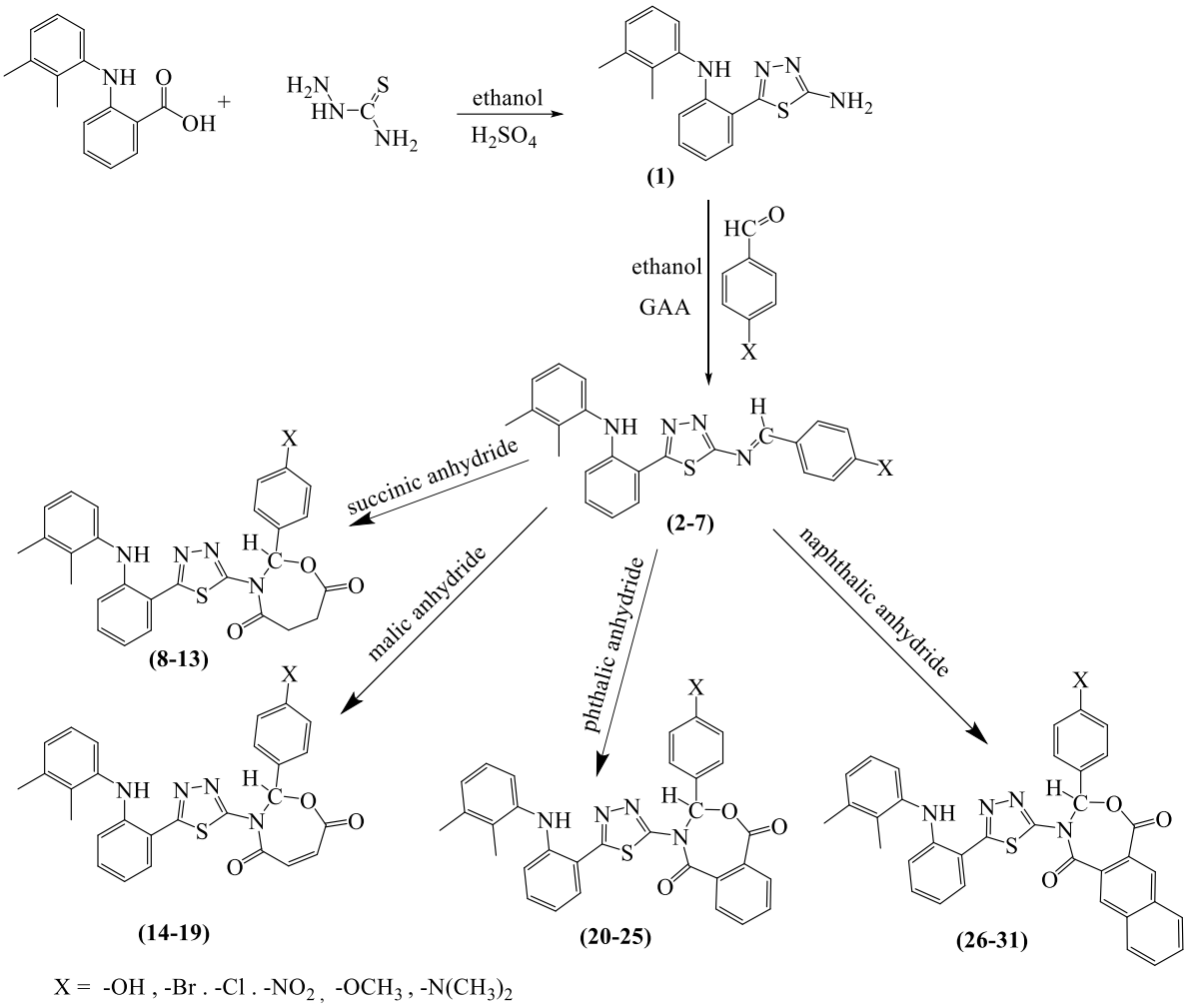
2. Experimental

2.1. Materials and Instruments

Sigma-Aldrich and GCC Chemicals were the suppliers of all chemicals and solvents. Uncorrected melting points were recorded using electro-thermal melting p0int equipment. The Shimadzu (IR Prestige 21) Fourier Transf0rm Infrared Spectr0meter's KBr discs were used t0 record the FTIR spectra. With TMS serving as an internal reference, 1HNMR spectra were captureed using DMSO-d6 and CDCl3 on a Bruker Ultra Shield (300) MHz instrument in Iran.

2.2. Synthetic Procedures

The routes f0r synthesizing new derivatives depend 0n the Scheme (1):



Scheme (1)

2.2.1. Preparation of 5-(2-((2,3-Dimethylphenyl) Amino) Phenyl)-1,3,4-Thiadiazol-2-Amine (1).

For 16 hours,(40 mL) of ethanol and (10.8 mL) of sulfuric acid were combined with equal m0les of mefenamic acid and thi0semicarbazide, and the mixture was refluxed. After that, the precipitate of compound (1) was dried and filtered. Production 85%, M.P. (119–121) °C. Lit. Yield 78%; M.P (115-120)ºC [2].

2.2.2. Synthesis of 2-[N-(4-Substituted Benzilidine] 5-(2-((2,3-Dimethylphenyl) Amin0) Phenyl)-1,3,4-Thiadiaz0l [2-7].

For five hours, compound (1) (0.014 mole, 4.2g) and aldehyde (0.014 mol) were refluxed in 40 mL of pure ethanol with drops of glacial acetic acid. After cooling the combination, the solid was filtered, separated from the ethanol by recrystallization, and collected.[22, 23] Table 1 lists the dried product's physical characteristics.

2.2.3. Synthesis of (Z)-2-(4-Substituted Phenyl)-3-(5-(2-((2,3-Dimethylphenyl) Amino) Phenyl)-1,3,4-Thiadiazol -2\-yl)-1,3-Oxazepin-4,7-Dione(8-31)

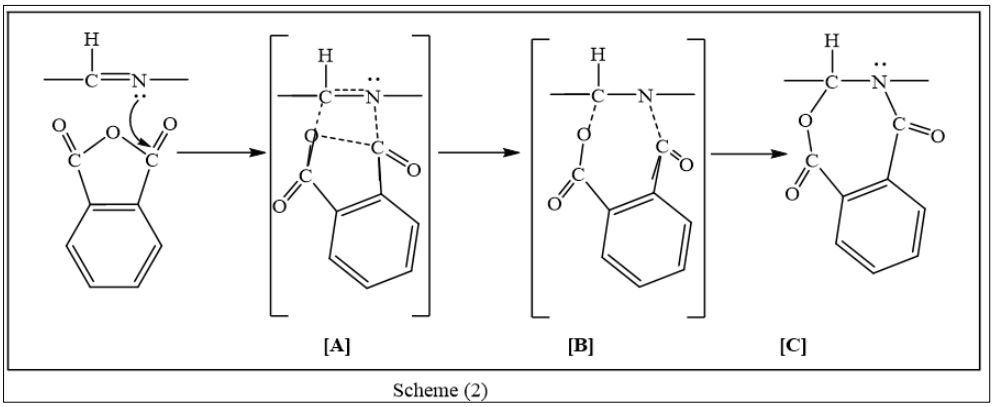
A mixture of Schiff base (2-7) (0.004 mole) with different anhydride [malic, succinic, phthalic, and naphthalic (0.004 mole)] was diss0lved in (25 mL) of dry benzene and then the mixture was refluxed f0r 7 hours then excess s0lvent

was distilled and the solids were filtered off and recrystallized from ethanol [24]. Physical properties of the dry product are listed in Table (1).

3. RESULTS AND DISCUSSION

The carboxylic acid group in mefenamic acid reacted with thiosemicarbazide in an acidic solution to create a number of new 1,3,4-thiadiazol-2-amines, which were then converted to compound (1) in good yield. The amino group's (NH2) nucleophilic assault started the process. A new absorption stretching band to (N=C) formed at 1642 cm^{-1}, and the stretching band asymmetry and symmetry of the N-H group appeared at (3340, 3309) cm-1. The FT-IR spectra for compound (1) also reveals new absorption stretching bands due to the (N-H) of the (NH_2) moiety in the area (3351,3162)cm^{-1}. By condensing compound (1) with various aldehydes in ethanol as a solvent and adding a drop of GAA, compounds (2–7) were created.

Mefenamic acid-based 7-membered cyclic ring 1,3-oxazepines-4,7-dione derivatives (8-31). By reacting a novel Schiff base (2-7) with five atoms of cyclic anhydride, such as succinic, maleic, phthalic, or naphthalic anhydride, in the presence of dry benzene as a solvent, the newly 1,3-oxazepine (8-31) was produced. A seven-membered heterocyclic ring is obtained by unlocking into acid anhydride after the σ bond of the C-O group of acid anhydride is added to the π bond of the imine group to produce a four-membered hetero ring and a five-membered cyclic ring in the same [A]. Compounds (8-31) [C], which have been thoroughly described and associated with their structures by spectroscopic investigation, provide a summary of the suggested mechanism. The suggested in Scheme (2) [26].



Scheme (2)

Table 1: Physical properties of the synthesized comp0unds (2-31)

Comp. No.	*Group X*	*Molecular Formula*	*Molecular Weight (g/mole)*	*Yield (%)*	*M.P (°C)*	*Colour*
2	-OH	$C_{23}H_{20}N_4OS$	400.50	92	102-104	White
3	-Br	$C_{23}H_{19}BrN_4S$	463.40	88	155-157	Pale yellow
4	-Cl	$C_{23}H_{19}ClN_4S$	418.94	78	144-146	White
5	$-NO_2$	$C_{23}H_{19}N_5O_2S$	429.50	82	171-173	Yellow
6	$-OCH_3$	$C_{24}H_{22}N_4OS$	414.53	87	138-140	White
7	$-N(CH_3)_2$	$C_{25}H_{25}N_5S$	427.57	92	166-168	Pale yellow
8	-OH	$C_{27}H_{24}N_4O_4S$	500.57	83	135-137	Yellow
9	-Br	$C_{27}H_{23}BrN_4O_3S$	563.47	81	188-190	Pale yellow
10	-Cl	$C_{27}H_{23}ClN_4O_3S$	519.02	73	169-171	Off White
11	$-NO_2$	$C_{27}H_{23}N_5O_5S$	529.57	79	192-194	Pale yellow
12	$-OCH_3$	$C_{28}H_{26}N_4O_4S$	514.60	83	165-167	White
13	$-N(CH_3)_2$	$C_{29}H_{29}N_5O_3S$	527.64	82	138-140	Yellow
14	-OH	$C_{27}H_{22}N_4O_4S$	498.57	76	130-132	Pale yellow
15	-Br	$C_{27}H_{21}BrN_4O_3S$	561.47	73	187-189	White
16	-Cl	$C_{27}H_{21}ClN_4O_3S$	517.02	68	166-168	Pale yellow
17	$-NO_2$	$C_{27}H_{21}N_5O_5S$	527.57	81	187-179	Off White
18	$-OCH_3$	$C_{28}H_{24}N_4O_4S$	512.60	76	160-162	Pale yellow
19	$-N(CH_3)_2$	$C_{29}H_{27}N_5O_3S$	525.64	85	134-136	Orange
20	-OH	$C_{31}H_{24}N_4O_4S$	542.68	72	144-146	White
21	-Br	$C_{31}H_{23}BrN_4O_3S$	611.51	79	204-206	Pale yellow
22	-Cl	$C_{31}H_{23}ClN_4O_3S$	567.06	74	192-194	White
23	$-NO_2$	$C_{31}H_{23}N_5O_5S$	577.62	80	230-232	Pale yellow

Comp. No.	*Group X*	*Molecular Formula*	*Molecular Weight (g/mole)*	*Yield (%)*	*M.P (°C)*	*Colour*
24	$-OCH_3$	$C_{32}H_{26}N_4O_4S$	562.64	87	181-183	White
25	$-N(CH_3)_2$	$C_{33}H_{29}N_5O_3S$	575.69	84	150-152	Pale green
26	-OH	$C_{35}H_{26}N_4O_4S$	598.68	65	154-156	Yellow
27	-Br	$C_{35}H_{25}BrN_4O_3S$	661.57	88	222-224	Dark yellow
28	-Cl	$C_{35}H_{25}ClN_4O_3S$	617.12	83	203-205	White
29	$-NO_2$	$C_{35}H_{25}N_5O_5S$	627.68	77	246-248	Pale green
30	$-OCH_3$	$C_{36}H_{28}N_4O_4S$	612.70	71	195-197	White
31	$-N(CH_3)_2$	$C_{37}H_{31}N_5O_3S$	625.75	85	174-176	Pale orange

3.1. FT-IR of Prepared Comp0unds

FT-IR spectral data are listed in Table (2). Prepared compounds (2-7) [25].

Table 2: The FT-IR characteristic bands of comp0unds (2-7)

Comp. No.	Changed part(x)	IR, KBr, υ, cm^{-1}					
		(N-H)	(C-H) Ar.	(C-H) Aliph.	(C=N)	(C=C) Ar.	Others Bands
2	OH	3345	3096	2999,2900	1610	1510,1579	O-H 3272
3	Br	3334	3090	2929,2881	1613	1587,1572	C-Br 684
4	Cl	3342	3034	2989,2841	1627	1554,1600	C-Cl 1020
5	NO_2	3356	3011	2972,2899	1605	1529,1606	C-NO_2 1390-1450
6	OCH_3	3330	2995	2945,2856	1673	1552,1604	C-O-C 1157
7	$N(CH_3)_2$	3335	3000	2991,2855	1620	1584,1591	N-(CH_3) 1288

FT-IR spectral data are listed in Table (3). Prepared compounds (8-31) [25].

Table 3: The FT-IR characteristic bands of comp0unds (8-31)

C0mp. No.	Changed part(x)	IR, KBr, υ, cm^{-1}					
		(C-H) Ar.	(C-H) Aliph.	(C=O)	(C-N)	(C-O-C)	Others Bands
8	-OH	3091	2987,2892	1690, 1729	1231	1165,1205	O-H 3272
9	-Br	3064	2902, 2978	1697, 1724	1234	1174,1199	C-Br 805
10	-Cl	3067	2978,2888	1695, 1714	1222	1158.1188	C-Cl 1020
11	$-NO_2$	3097	2966, 2985	1633, 1699	1228	1163,1228	C-NO_2 1375,1448
12	$-OCH^3$	3003	2950,2877	1682,1714	1235	1150,1236	
13	$-N(CH_3)_2$	3100	2899, 2950	1641, 1720	1236	1101,1216	N-CH_3 1236
14	-OH	3098	2988,2887	1692, 1726	1238	1158,1211	O-H 3272
15	-Br	3057	2976, 2816	1697, 1724	1311	1172	C-Br 702
16	-Cl	3067	2978,2888	1695, 1714	1222	1158.1188	*C*-Cl 1020
17	$-NO_2$	3090	2850, 2925	1674, 1711	1288	1290	C-NO_2 1376,1488
18	$-OCH_3$	3008	2961,2870	1680,1711	1230	1152,1238	
19	$-N(CH_3)_2$	3039	2914, 2870	1688, 1720	1295	1168	N-CH3 1295
20	-OH	3092	2984,2892	1690, 1727	1231	1165,1205	O-H 3282
21	-Br	3059	2793,2970	1678,1693	1165	1260,1361	C-Br 812
22	-Cl	3069	2985,2878	1689, 1711	1222	1158,1188	C-Cl 1031
23	$-NO_2$	3070	2937,2978	1697,1728	1180	1199,1065	C-NO_2 1350-1419
24	$-OCH_3$	2992	2941,2870	1684,1708	1231	1158,1233	
25	$N(CH_3)_2$	3036	2908,2958	1657,1710	1170	1213,1077	N-CH_3 1265

C0mp. No.	Changed part(x)	IR, KBr, υ, cm^{-1}					
		(C-H) Ar.	(C-H) Aliph.	(C=O)	(C-N)	(C-O-C)	Others Bands
26	-OH	3099	2996,2894	1693, 1728	1235	1163,1204	O-H 3277
27	-Br	3058	2978, 2819	1695, 1730	1319	1169	C-Br 722
28	-Cl	3085	2971,2884	1690, 1709	1218	1152,1183	C-Cl 1024
29	$-NO_2$	3075	2931,2972	1692,1724	1182	1194,1062	C-NO_2 1351-1418
30	$-OCH_3$	2989	2945,2870	1672,1711	1229	1151,1231	
31	$-N(CH_3)_2$	3039	2915,2963	1655,1708	1172	1211,1073	N-CH_3 1261

3.2. 1H-NMR of Some Prepared Comp0unds

1H-NMR spectrum of the compound [4], showed the following signals: the appearance of a signal at δ (9.35) ppm, to be ascribed to (N-H) moiety, the appearance of a signal at chemical shift δ (8.22) ppm due to a proton (H-C=N), a multiplet signal at δ (6.74-8.11) ppm ascribed to (1H) from the benzene rings. Additionally, signals were observed at chemical shifts δ (2.15) ppm and δ (2.32) ppm, which could be assigned to the (CH_3) prot0ns of the two groups.

The compound's (1H-NMR) spectrum [8], displayed the following signals: A signal that might be attributed to N-H appeared at δ (9.23) ppm, a signal that belonged to a proton (H-O) occurred at chemical shift δ (8.77) ppm, and a signal that belonged to a proton (H-C-N) appeared at chemical shift δ (8.12) ppm. A multiplet signal was 0bserved at δ (6.82-7.88) ppm, which was caused by (1H) and could be attributed to the benzene rings. The oxazipene ring's δ (CH2) protons caused a signal at δ (2.24) ppm and a signal at δ (2.56) ppm. The two groups of (CH3) protons caused a signal at δ (2.13) ppm and δ (2.27) ppm.

The (1H-NMR) spectrum of comp0und [23], showed the following signals: A signal appeared at δ (9.51) ppm which could be assigned to (N-H), the appearance of a signal at chemical shift δ (8.33) ppm belonging to a proton (H-C-N), A multiplet signal at δ (6.95-8.11) ppm due to (1 H) that could be assigned to the benzene rings, showed a signal at the chemical shift δ (2.32) ppm and δ (2.45) ppm a signal, due to the two groups (CH_3) protons.

3.3. 13C-NMR of Some Prepared Compounds

A signal at the chemical shift (19.15,21.88) ppm of carbon(CH3), two groups, and a signal at the chemical shift (85.48) ppm due to carbon (O-C-N) were observed when examining the (13C-NMR) of compound (23). There were also multiple signals at the chemical shift (113.64-153.35) ppm belonging to Carbon (C=C) for aromatic rings and a signal at the chemical shift (171.25,172.78,) ppm of the two signals referring to carbon (C=O).

When examining the (13C-NMR) of compound (23), it was observed that a signal for carbon (CH3) two groups appeared at the chemical shift (20.23,23.14) ppm, a signal for carbon (CH2) two groups appeared at the chemical shift (42.78,45.90) ppm, and a signal for carbon (O-C-N) at the chemical shift (89.10) ppm. There were also multiple signals at the chemical shift (112.68-150.45) ppm that belonged to Carbon (C=C) in aromatic rings, and a signal at the chemical shift (169.56, 170.41) ppm, corresponding to the two signals, corresponded to carbon (C=O).

When examining the (13C-NMR) of compound (23), it was observed that several signals at the chemical shift (111.17-151.97) ppm belonging to Carbon (C=C) for aromatic rings, a signal at the chemical shift (167.72,169.13,) ppm of the two signal ppm refers to Carbon (C=O), and a signal at the chemical shift (22.12,24.34) ppm belonging to carbon(CH3) two groups and a signal at the chemical shift (91.84) ppm due to carbon (O-C-N).

4.0. Biological Activity

In the present study, an attempt was made to synthesize various derivaitives of the substaituted 1,3,4-thiadiazole ring based on a new oxazepine derivative moiety (from mefenamic acid) and to assess their biological evaluation of antibacterial activity. Heterocyclic compounds are a significant class of organic chemicals due to their use in pharmaceuticals and industrial research. Antibacterial medications work by inhibiting cell wall production, protein synthesis, nucleic acid synthesis, metabolic pathway inhibition, and interference with cell membrane integrity. Compared to aspirin, mefenamic acid has three times the analgesic effect. Nevertheless, they are linked to a higher risk of stomach ulcers, much as other traditional NSAIDs. In this work, 1,3,4-thiadiazole and Schiff base, as well as their novel oxazepine derivatives—which are anticipated to have less adverse effects than the original compound—are synthesized and their antibacterial, cytotoxic, and in vivo activities are evaluated [27–33].

4.1. Antibacterial Activity

Using the agar diffusion technique, the compounds' antibacterial activity was investigated (in vitro) against Staph. Aureus (G+), Bacillus subtilis (G+), Klebsiella pneumoniae (G-), and E. coli (G-). According to Table 4, the majority of the derivatives exhibit either strong or modest biological activity against bacteria. Due to the presence of 1,3,4-thiadiazole, compounds (23,28) showed excellent inhibition against Staphylococcus aureus, whereas compounds (15,20) had minor activity against E. coli and Klebsiella pneumonia.

Table 4: antibacterial activity of some compounds

***No.* Comp.**	***Zone of inhibition in millimeter***			
	Staphyl ococcus aureus	***Bacillus sabtilis***	***Klebsiella pneumoniae***	***Escherichis coli***
9	19	18	17	21
11	11	5	12	11
15	13	5	18	6
19	22	18	15	12
20	17	12	5	6
23	25	23	21	19
25	21	19	12	14
26	15	17	13	13
29	27	24	23	22

5. CONCLUSIONS

Lastly, the synthesis pathway for novel 1,3oxazepine derivatives based on mefenaimic acid is given. These derivatives should have the biological activity to achieve improved action and fewer adverse effects in the stomach. The presence of the oxazepaine ring with the NO_2 group in the molecule structure may be the reason why compounds (23) and (29) exhibit antimicrobial activity (in vitro). As a result, they have the potential to be extremely promising starting comp0unds for the development of a new pharmaceutaical drug beyond pharmacological projects and lessen the more negative effects of mefenamic acid.

Acknowledgement: This work was funded by Al-Nahrain University's Bio Technology Research Center.

Funding: A particular grant from a governmental, private, or nonprofit funding organization was obtained for this study.

Contribution of the Author: The author consented to be in charge of all facets of this study and contributed to the data analysis, drafting, and revision of the manuscript.

Conflict of Interest: There are no conflicts of interest, according to the author.

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