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Synthesis of Thiazolidine Derivatives from Schiff bases and study their as Antibacterial Effect

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ABSTRACT

This research includes the synthesis of some new heterocyclic derivatives (pyridine derivatives), firstly reaction of (2-amino-5-chloropyridine, 2-amino-4-methyl pyridine) With aromatics benzaldehydes (p-bromobenzaldehyde, dimethyl amino benzaldehyde , p-chlorobenzaldehyde, Salicylic aldehyde, p-amino benzaldehyde) to form different derivatives of schiff bases, secondly reaction of Schiff base derivatives with thioglycolic acid to form thiazolidinone derivatives, all resulting compounds characterized by FT-IR.

Keywords: Anti-Bacterial activity, Pyridine, Thiazolidinone, Schiff bases.

1. Introduction

Hetero cyclic compounds are a group of important compounds that contain atoms different from the carbon atom [1], it has antibacterial [2], anti-cancer [3], anti-inflammatory [4],activity so it's included in composition of many important compounds such vitamins, heamogloben, purines and pyrimidines. It attracted a lot of attention because it is considered the basic nucleus of many pharmaceutical compounds such Acyclovi , Clopidogrel, Pilocarpine, Mimosine.

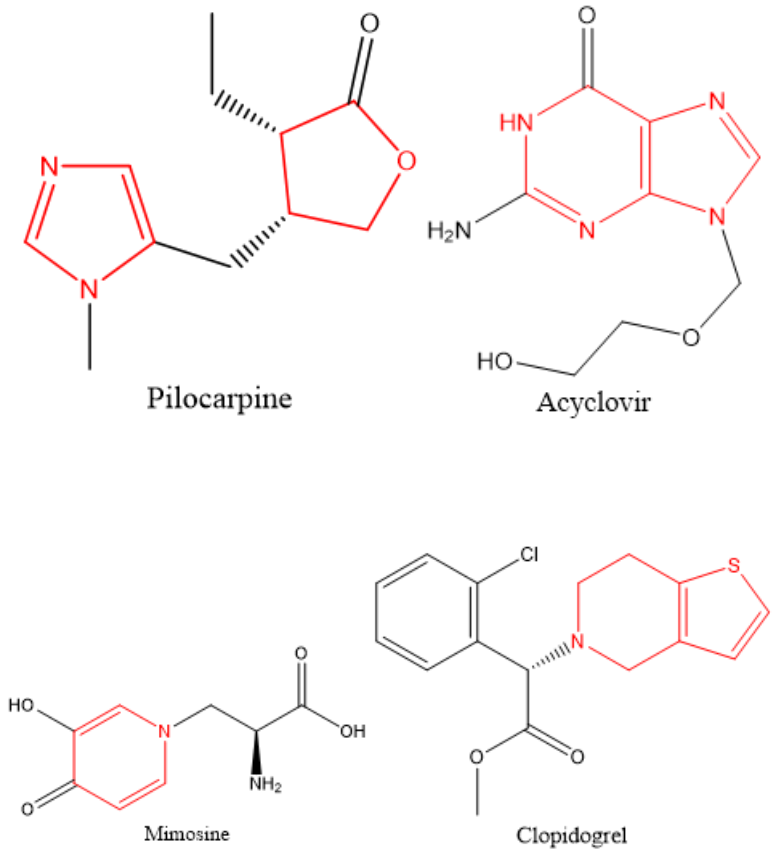


Figure 1. Shows the structure of some drug containing heterocycle rings

Thiazolidine is a class of heterocyclic compounds that are characterized by the presence of sulfur and nitrogen as heterocyclic atoms in aromatic ring, thus the molecular formula of the thiazolidine ring is C3H7NS [5], if the thiazolidine ring contains carbonyl group it is called thiazolidinone. The presence of both Nitrogen and sulfur atoms in the same ring enhances their importance in many medical and pharmaceutical fields, where thiazolidine compound consider essential components of many naturals products and manufactured drugs so it has great anti-viral, anti-microbial, biological importance as it is used as antitumor, antibiotics, Anti-Trypanosoma, anti oxidant, analgesic, anti-inflammatory, anti-cancer, antifungal, Anticonvulsants, Antitubercular, against γ -Irradiation, anti-diabetic, reduce hyperlipidaemia, anti malaria, and anti HIV-1 [6-22].

2. Materials and Methods

2.1. Materials

FT-IR Spectra (400 -4000 cm-1) in KBr disk were recorded on SHIMADZU FTIR-8400S Fourier transform.

Melting points were measured using Stuart.

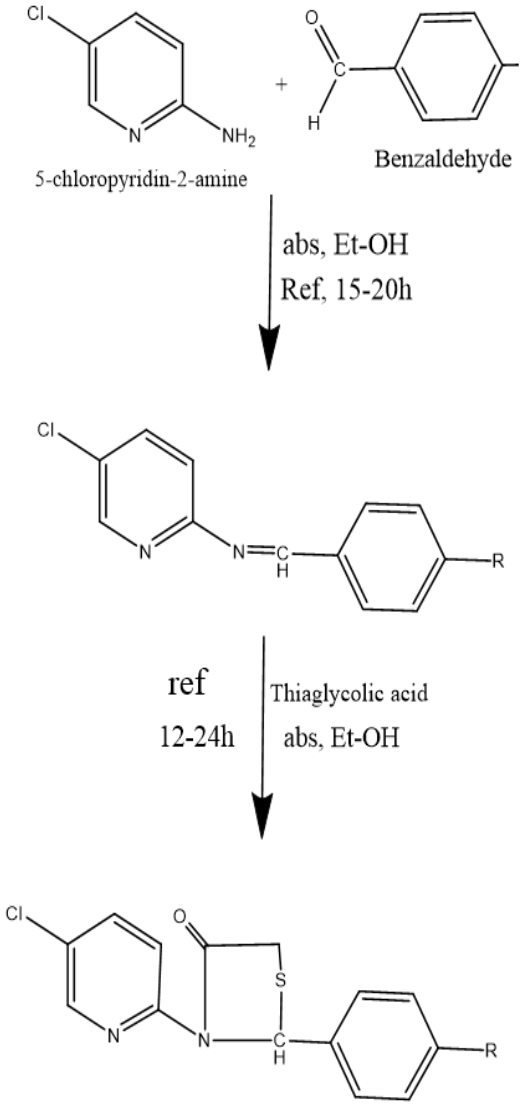
2.2. Imine preparation

We put a certain amount of the amino pyridine derivatives with a certain balanced amount of aldehyde compounds and dissolving each separately with an appropriate amount of ethanol after the complete dissolution in the ethanol the two compounds were mixed and the addition of drops of glacial acetic acid

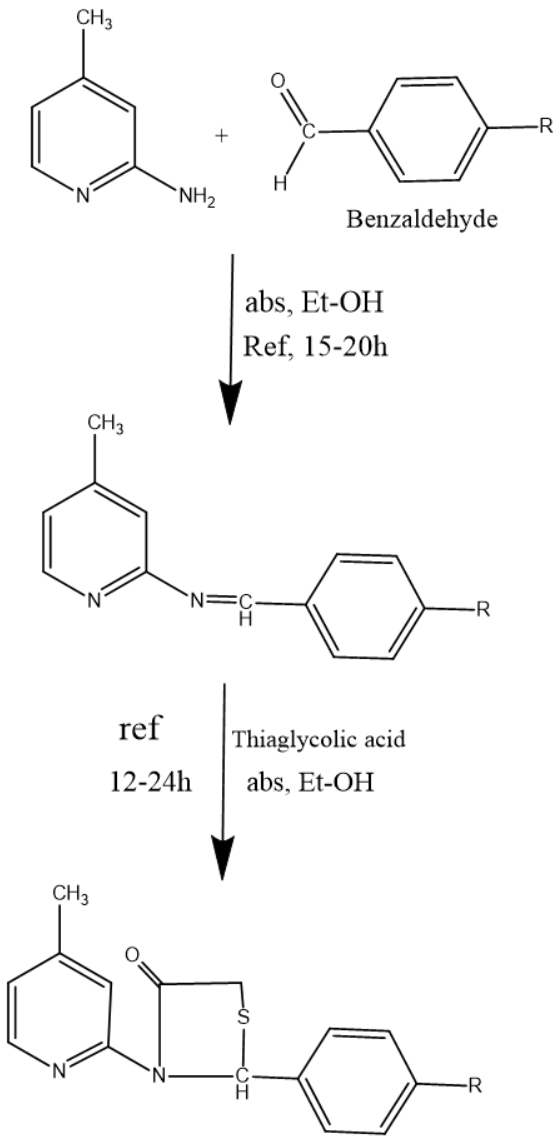
and the escalation process for the prepared mixture and after completing the escalation process the mixture was left to cool and during the process the reaction was monitored by TLC technique using methanol and dry benzene t by 1:4 as an eluent [22].

2.3. Thiazolidinones preparation

Equal amounts of Schiff base derivatives (0.01 mol) and thioglycolic acid (0.01 mol, 0.20 ml) in an absolute ethanol (50) ml were refluxed for 10-30 h. The progress of reaction was monitored by TLC using hexane and ethyl acetate (3:7) as an eluent. After reaction complete, the solvent (ethanol) was removed to give a solid compound which was dissolved in hot ethanol [23].



Scheme 1. synthesis of thiazolidinone derivatives using 5-chloro-2-amino pyridine as a starting material



Scheme 2. synthesis of thiazolidinone derivatives using 4-methyl-2-amino pyridine as a starting material

3. Results

3.1 FT-IR spectrum data for compounds 1-10

FT-IR spectrum data for compound 1 show band at 1664 cm-1 for C=N , 1597 CM-1 for C=C , 3095 cm-1 for C-H aromatic, 1165 cm-1 for C-N , 821 cm-1 for C-Cl.

FT-IR spectrum data for compound 2 show band at 1664 cm-1 for C=N , 1597 CM-1 for C=C , 3091 cm-1 for C-H aromatic, 1165 cm-1 for C-N , 823 cm-1 for C-Cl.

FT-IR spectrum data for compound 3 show band at 1622 cm-1 for C=N , 1570 CM-1 for C=C , 3080

cm-1 for C-H aromatic, 1109 cm-1 for C-N , 840 cm-1 for C-Cl.

FT-IR spectrum data for compound4 show band at 1620 cm-1 for C=N ,1571 cm-1 for C=C , 3080 cm-1 for C-H aromatic, 1107 cm-1 for C-N , 823 cm-1 for C-Cl , 565cm-1 for C-Br

FT-IR spectrum data for compound 5 show band at 1662 cm-1 for C=N ,1597 cm-1 for C=C 3300 cm-1 for N-H , 2910 cm-1 for C-H aromatic, 1165 cm-1 for C-N.

FT-IR spectrum data for compound 6 show band at 1614 cm-1 for C=N ,1566 cm-1 for C=C, 3080 cm-1 for C-H aromatic, 1176 cm-1 for C-N,798 cm-1 for C-Cl.

FT-IR spectrum data for compound 7 show band at 1664 cm-1 for C=N ,1597 cm-1 for C=C, 3091 cm-1 for C-H aromatic, 1165cm-1 for C-N,594 cm-1 for C-Br.

FT-IR spectrum data for compound 8 show band at 1666 cm-1 for C=N ,1597 cm-1 for C=C, 3045 cm-1 for C-H aromatic, 1165 cm-1 for C-N.

FT-IR spectrum data for compound 9 show band at 1610 cm-1 for C=N ,1570 cm-1 for C=C, 3076 cm-1 for C-H aromatic, 1184 cm-1 for C-N , 1278 cm-1 for C-O.

FT-IR spectrum data for compound 10 show band at 1668 cm-1 for C=N ,1597cm-1 for C=C, 3053cm-1 for C-H aromatic, 1145cm-1 for C-N , 1271 cm-1 for C-O, 785 cm-1 for C-Cl.

Table 1. FT-IR spectrum data for compounds 1-10.

OTHERS	C-N	C-H (aromatic)	C=C	C=N	compound
821 cm-1 for C-Cl ,	1165	3095	1597	1664	1
823 cm-1 for C-Cl	1165	3091	1597	1664	2
840 cm-1 for C-Cl	1109	3080	1570	1622	3
823 cm-1 for C-Cl , 817cm-1 for C-Br	1107	3080	1571	1620	4
3300 cm-1 for N-H	1165	2910	1597	1662	5
798 cm-1 for C-Cl	1176	3080	1566	1614	6
594 cm-1 for C-Br	1165	3091	1597	1664	7
	1165	3045	1597	1666	8
1278 for C-O	1184	3076	1570	1610	9
For C-O 1271 For C-Cl 785	1145	3053	1597	1668	10

The disappearance of absorption beams of the NH2 group in the range (3433-3300 cm-1) were observed.

3.2 Physical properties of compound (1-10)

Table 2. Physical properties of compound (1-10).

Reaction time	Rf	color	Yield %	m.p	compound
15h	0.7	Light brown	54%	70-73	1
17h	0.7	Yellow	60%	71-74	2
20h	0.6	White	48%	151-153	3
20h	0.6	White	40%	167-169	4
18h	0.7	Orange	56%	68-70	5
20h	0.8	White	66%	113-116	6
19h	0.7	White	51%	122-124	7
17h	0.8	Dark brown	68%	50-52	8
20h	0.5	Yellow	%61	127-129	9
18h	0.6	Orange	70%	73-75	10

3.3 FT-IR spectrum data for compound (11 - 20)

FT-IR spectrum data for compound 11 show band at 1660 cm-1 for C=O, 1597 cm-1 for C=C , 2916 cm-1 for C-H aromatic, 1166 cm-1 for C-N , 823 cm-1 for C-Cl , 729 cm-1 for C-S

FT-IR spectrum data for compound 12 show band at 1666 cm-1 for C=O, 1598 cm-1 for C=C , 2980 cm-1 for C-H aromatic, 1168 cm-1 for C-N , 823 cm-1 for C-Cl , 729 cm-1 for C-S , 3452 cm-1 for N-H

FT-IR spectrum data for compound 13 show band at 1689 cm-1 for C=O, 1593 cm-1 for C=C , 3070 cm-1 for C-H aromatic, 1141 cm-1 for C-N , 827 cm-1 for C-Cl , 663 cm-1 for C-S

FT-IR spectrum data for compound 14 show band at 1691 cm-1 for C=O, 1568 cm-1 for C=C , 3082 cm-1 for C-H aromatic, 1109 cm-1 for C-N , 840 cm-1 for C-Cl ,817 cm-1 for Br ,700 cm-1 for C-S

FT-IR spectrum data for compound 15 show band at 1664 cm-1 for C=O, 1597 cm-1 for C=C , 3305 cm-1for N-H , 2902 cm-1 for C-H aromatic, 1166 cm-1 for C-N ,690 cm-1 for C-S

FT-IR spectrum data for compound 16 show band at 1666 cm-1 for C=O, 1597 cm-1 for C=C , 3039 cm-1 for C-H aromatic, 1165 cm-1 for C-N , 815 cm-1 for C-Cl ,727 cm-1 for C-S

FT-IR spectrum data for compound 17 show band at 1666 cm-1 for C=O, 1566 cm-1 for C=C , 3018 cm-1 for C-H aromatic, 1176 cm-1 for C-N , 798 cm-1 for C-Br ,686 cm-1 for C-S

FT-IR spectrum data for compound 18 show band at 1664 cm-1 for C=O, 1598 cm-1 for C=C , 3039 cm-1 for C-H aromatic, 1166 cm-1 for C-N,690 cm-1 for C-S

FT-IR spectrum data for compound 19 show band at 1668 cm-1 for C=O, 1571 cm-1 for C=C , 3037 cm-1 for C-H aromatic, 3323 cm-1 for OH , 1180 cm-1 for C-N,790 cm-1 for Cl ,692 cm-1 for C-S

FT-IR spectrum data for compound 20 show band at 1672 cm-1 for C=O, 1570 cm-1 for C=C , 3078 cm-1 for C-H aromatic, 3309 cm-1 for OH , 1188 cm-1 for C-N,790 cm-1 for Cl ,665 cm-1 for C-S

Table 3. FT-IR spectrum data for compound (11 - 20)

Others	C-S cm-1	C-N cm-1	C-H (aromatic) cm-1	C=C cm-1	C=O cm-1	Compound
823 cm-1 for C-Cl	729	1166	2916	1597	1660	11
3452 cm-1 for N-H, 823 cm-1 for C-Cl	729	1168	2980	1598	1666	12
827 cm-1 for C-Cl	663	1141	3070	1593	1689	13
840 cm-1 for C-Cl ,817 cm-1 for Br	700	1109	3082	1568	1691	14
3305 cm-1for N-H	690	1166	2902	1597	1664	15
815 cm-1 for C-Cl	727	1165	3039	1597	1666	16
798 cm-1 for C-Br	690	1176	3018	1566	1666	17
	690	1166	3039	1598	1664	18
790 cm-1 for Cl, 3323 cm-1 for OH,	692	1180	3037	1571	1668	19
790 cm-1 for Cl, 3309 cm-1 for OH,	665	1188	3078	1570	1672	20

The disappearance of absorption beams of the C=N (azomethengroup) were observed.

3.4 Physical properties of compound (11-20)

Table 4. Physical properties of compound (11-20)

Reaction time	Rf	color	Yield %	m.p	compounds
24h	0.4	Dark brown	65%	95-98	11
26h	0.9	Light brown	67%	50-53	12
24h	0.8	Dark yellow	55%	97-100	13
23h	0.7	Light yellow	55%	166-169	14
20h	0.8	Dark brown	67%	50-53	15
23h	0.6	Dark yellow	72%	94-96	16
24h	0.9	Dark yellow	68%	101-104	17
20h	0.7	Dark brown	58%	65-68	18
22h	0.9	Dark brown	73%	77-79	19
22h	0.9	Light yellow	77%	102-105	20

3.5 Biological activity

The effect of the prepared compounds on some types of bacteria, namely (S.aureus and E.coli) was studied, as shown in Table 5

Table 5. the effect of prepared compounds against two types of bacteria (E. Coli and S.Aureus)

E.coli	S.aureus	compounds
Inhibition diameter(millimeter) after 24 h		
3.3	3	1
3.1	3	2
3	3	3
3.5	3.5	4
3	3.1	5
3.2	3	6
3.1	3.4	7
3.1	3	8
3	3.2	9
3.5	3.2	10
3.2	3.5	11
3	3.5	12
3	3	13
3.1	3.2	14
3.5	3.5	15
3.1	3.1	16
3	3.5	17
3.2	3	18
3.5	3.4	19
3.5	3	20

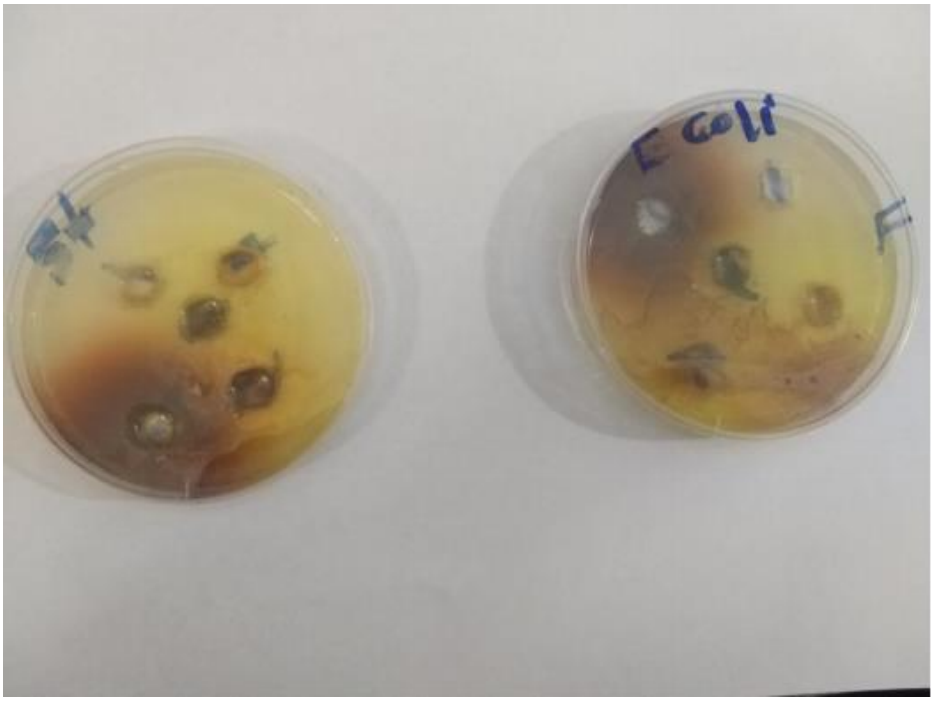


Figure 2. the effect of the prepared compounds against the S.aureus and E.coli bacteria

4. Discussion

It was observed at (FT-IR) data for (1-10) compounds the appearance of azomethengroup (C=N) and disappearance of NH2 group in the range (3433-3300 cm-1) indicate the formation of Schiff base derivatives , then after formation of Thiazolidinone derivatives (FT-IR) data shown the disappearance of azomethengroup (C=N) and appearance of the appearance of (C=O) at (1660-1700) cm−1 and (C-S) at (650-770)cm−1 mean the formation of thiazolidine derivative.

5. Conclusion

AS it was shown during preparing the compounds that the difference in the groups substituted on the same compound leads to a difference in the results, reaction time and biological activity of the resulting compounds. The study of biological activity of Schiff base derivatives and thiazolidine derivatives against E. Coli show that all derivatives give a positive results at different diameter but the compounds 3,5,9,12,13,17) give a lower inhibition and the compounds (4,10,15,19,20) give higher inhibithion. The compounds (1,2,3,6,8,13,18,20) give lower inhibition against S.

Aureus as well as the compounds (4,11,12,15,17) give higher inhibition. In FT-IR spectrum data for (1,2,3,4,5,6,7,8,9,10) compounds it's clearly the disappearance of NH2 peak at (3433-3300 cm−1) and appearance azomethen group (1610–1666 cm−1) mean formation of Schiff base derivatives. At (11,12,13,14,15,16,17,18,19,20) compounds it's clearly the appearance of (C=O) at (1660-1700) cm−1 and (C-S) at (650-770)cm−1 mean the formation of thiazolidine derivative.

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