

Synthesis, Characterization, Biological Evaluation and Anti Corrosion Activity of some Heterocyclic Compounds Oxazepine Derivatived from Schiff Bases

Khuluod Fahed Hamak^{1*}, Hamid Hussein Eissa²

**Chemistry Department, College of Science – Damascus University,
Damascus, Syria
Chemistry Department, Education and Applied College Sciences,
University of Hajah, Yemen**

**Corres. author: khlood.hamak@yahoo.com*,
hamedesia2003@yahoo.com**

Abstract: A series of Schiff base and their derivative (oxazepine) have been synthesized 1,4-Bis (3-aminopropyl)-piperazine was condensed with various aromatic aldehyde in ethanol in the presence of acetic acid as catalyst to yield the Schiff base(1-8). These Schiff's bases on treatment with phthalic anhydride gave substituted oxazepine(9-16). The structure of synthesized has been established on the basis of their spectral (FT-IR, Mass, ¹H, ¹³C-NMR, elemental analysis) data. The purity of the compounds was confirmed by TLC. All these compounds were evaluated for their In vitro activity against several microbes .these compounds were tested to determine their ability to inhibit corrosion of mild steel in 1 mol.l⁻¹ H₂SO₄ .

Key words : Schiff bases, oxazepine ,antibacterial activity, anticorrosion,mild steel.

Introduction

The development of simple synthesis route to widely used organic compounds ring, using readily available reagents is one of the main objective of organic synthesis, Nitrogen heterocycles are of a special interest because they constitute an important class of natural and non natural products, many of which exhibit useful biological activities, one-pot efficient synthesis of heterocyclic derivatives, may permit the development of novel therapies for the treatment of epilepsy, pain and other neurodegen disorder¹.

Some Schiff bases bearing aryl groups², or heterocyclic residues possess excellent biological activities³, which has attracted many researcher's attention in recent year. They have been reported to used as analgesic, anthelmintic, antituberculer, plant growth regulator, antiviral, antifungal and anticancer⁴. Oxazepine (benzodiazepine) derivative introduced in 1965 for use in relief of the psychoneuroses characterized by anxiety and tension, oxazepam is non-homologous seven membered ring that contains two hetero atoms (oxygen and nitrogen)⁵. Oxazepine compounds have medical and biological important and they have medicinal and pharmaceutical application Among the wide Chemical derivatives are a heteropolymer which have activity And

effectiveness against cancer⁶ they also have effective against fungi and bacteria⁷, found that some Oxazepine derivative is considered a medical drug against the disease⁸ and also that (dibenzoxazepine) mental depression as in derivative Is a cyclic compound contains a heterogeneous (Amoxapine) compound, Oxazepine derivatives are found to be effective against anxiety and Associated with schizophrenia⁹ also found that 7-hydroxyamoxapine) is a pharmaceutical composite affecting the nervous center (CNS)¹⁰. A send, in (as example) is an antidepressant with a mild sedative component to its action in animals (omoxazepine) reduced the uptake of noradrenalin and serotonin and blocked

the response of dopamine receptors to dopamine¹¹. These interesting biological activities attracted our attention to the chemistry of nitrogen heterocycles. Some oxazepine derivatives act as inhibitors of some enzymes action¹².

Several Schiff bases have recently been investigated as corrosion inhibitors for various metals and alloys in acid media. These substances generally become effective by adsorption on the metal surface. The adsorbed species protect the metal from the aggressive medium, which causes decomposition of the metal. But also on the chemical structure of the inhibitor¹³⁻¹⁶.

In this work the inhibiting action of Schiff bases and their derivative on the corrosion steel in 1M H₂SO₄ solution has been investigated. The electrochemical techniques such as polarization measurements were used in this study. Differences in behavior of inhibitors were explained based on structural properties of investigated inhibitors.

Material and Methods

1-General Procedures:

Melting points were determined in open glass capillaries on a Gallenkamp apparatus and are uncorrected. TLC was performed to assess the reactions and the purity of the products. IR spectra were recorded in KBr (pellet forms) on a Nicolet-Avatar-330 FT-IR spectrophotometer and noteworthy absorption values (cm⁻¹) alone are listed. ¹H and ¹³C NMR Spectra were recorded at 400 MHz Bruker AMX using CDCl₃ as solvent. The ESI+ve MS spectra were recorded on a Bruker Daltonics LC-MS Spectrometer. Satisfactory microanalysis was obtained on Carlo Erba 1106 CHN analyzer. Potentiostat – galvanostat from Amel instruments were used for corrosion activity.

Chemical and starting materials

5-bromosalicylaldehyde, anthracene-9-carbaldehyde, 3-hydroxybenzaldehyde, 4-hydroxybenzaldehyde, O-vanillin, 4-chlorobenzaldehyde, 4-methylbenzaldehyde, 4-(dimethylamino)benzaldehyde and *N,N'*-bis(3-aminopropyl) piperazine (all from Aldrich) were used as supplied, without further purification.

2-General procedure for synthesis of Schiff base and its derivatives:

I. Preparation of Schiff bases (1-8)

A series of Schiff bases were prepared from the reaction of 1,4-Bis (3-aminopropyl)-piperazine (1 mole), with different aldehydes (2 moles), in 20ml ethanol absolute and few drops of glacial acetic acid. This mixture was refluxed for 2hrs. The mixture was cooled; precipitate was obtained then recrystallized from dioxane¹⁷.

II. Preparation of Oxazepine (9-16)

A mixture of Schiff base (0.0012 mole) and phthalic anhydride (0.0025 mole) was dissolved in (20mL) dry benzene. The mixture was heated for 5hrs in water bath at (70 C), excess solvent was distilled, the precipitate was filtered and recrystallized from ethanol¹⁸⁻¹⁹.

3. Biological Activity:

All newly synthesized compounds were tested for their in vitro growth inhibitory activity against a standard strain of pathogenic microorganism including Gram –positive bacteria (*Staphylococcus aureus*), Gram - negative bacteria (*Escherichia coli*, *Bacillus*) Antibacterial activity was done by the disk diffusion method. *S. aureus* and *E. coli* were sub cultured in BHI medium and incubated for 18h at 37⁰c, and then the bacterial cells were suspended, according to the McFarland protocol in saline solution to produce a suspension of about 10⁵CFU ml⁻¹: 10 μ of this suspension was mixed with 10 ml of sterile antibiotic agar at 40⁰C and poured onto an agar plate in a laminar flow cabinet. Five paper disks (6.0mm diameter) were fixed onto nutrient agar plate. 1 mg of each test compound was dissolved in 100 μ DMSO to prepare stock solution from stock solution different concentration 100, 250, 500, 1000 ppm of each test compound were prepared. These compounds of different concentration were poured over disk plate on to it. Streptomycin was used as standard drug (positive control) DMSO poured disk was determined by the formation of an inhibitory zone after 24h of incubation at 36⁰C. (Table 1) reports the inhibitory zones (mm) of each compound and the controls²⁰.

4. Anticorrosion Activity:

4.1. Electrochemical measurements: Were carried out in conventional three –electrode system in CHI 604 instrument (USA) at 303 K. The working electrode (mild steel) has a geometric area of 1 cm². The saturated calomel and platinum electrodes were used as reference and auxiliary electrodes. Equation (1) shows the calculation of IE from corrosion current: ²¹⁻²⁴

$$IE = \left(1 - \frac{I_{corri}}{I_{corr0}}\right) \times 100 \quad (1)$$

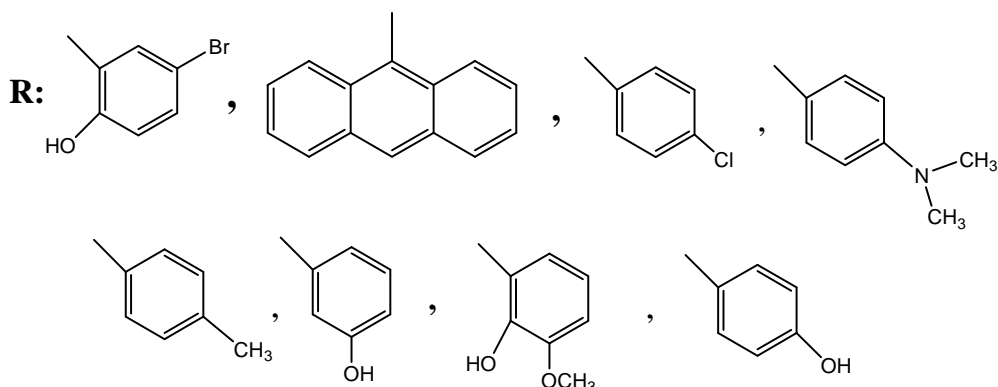
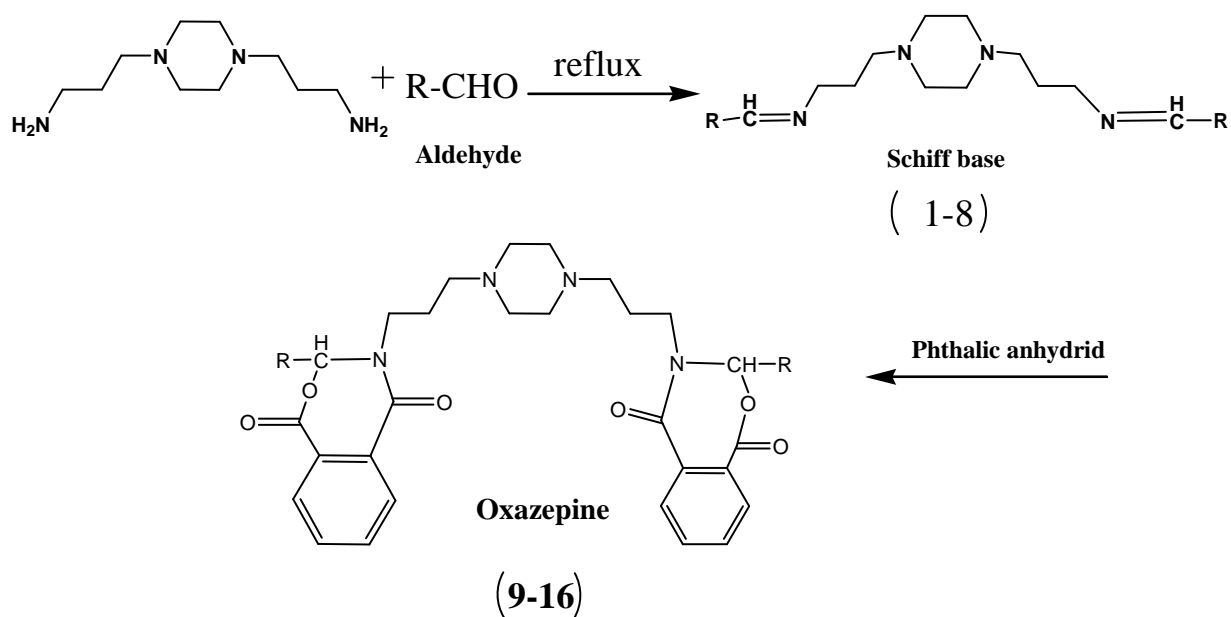
5. Results and Discussion :

5.1. Chemistry and characterization :

The present work involved two steps

First step : include preparation of new eight Schiff bases (1-8) were prepared by reaction of 1,4 bis (3-aminopropyl)-piperazine with different aldehydes. The synthesis of these compounds was carried out lined in Scheme (1) and the physical properties for Schiff base (1-6) including melting point range of (100-170) and % yield were range of (70-80) and these compounds were identified by FT-IR Spectroscopy, LC-MS, ¹H-, ¹³C-NMR. FT-IR spectrum of compounds (1-6) showed characteristic absorption bands (1635) cm⁻¹, (3140) cm⁻¹, (2885-2997) cm⁻¹ due to ν(C=N), ν(C-H) aromatic, ν(C-H) aliphatic respectively. as shown in table (3) [25,26] ¹H-NMR spectrum of compound (7) showed multiplet signals at (6.79-7.31)ppm due to aromatic protons and singlet signal at (8.20)ppm due to (C-H) group and singlet signal at 13.49 ppm due to (OH) group in addition to multiplet signals at (1.81) due to (C-H(9)) and multiplet signals at (2.33-2.40)ppm due to (C-H(11)), (C-H(10)) and triplet signal at (3.58) ppm due to (C-H(8)) ¹H-NMR of compound(8) show multiplet signals at (7.39, 7.91, 8.55 ppm) due to aromatic protons of anthracene

Second step : The second step included preparation of new eight Oxazepine (9-16) were prepared by reaction of Schiff bases (1-8) in (First step) with phthalic anhydride in dry benzene. The synthesis of these compounds was carried out lined in scheme (1). and the physical properties for oxazepine(9-16) including melting point range of (150-186)C⁰ and % Yield were range (70-80) and these compounds were identified by FT-IR, LC-MS and ¹H-, ¹³C-NMR. FT-IR spectrum of compounds (9-16) showed clear absorption bands at (1634.38)cm⁻¹ and (1709.59)cm⁻¹ attributed to the ν(C=O) of lactone and lactam inside oxazepine ring. The ¹H-NMR spectrum of compound (9-16), showed multiplet signals at (7.62-8.15) ppm due to aromatic protons of oxazepine ring and a singlet signal at (7.35)ppm due to N-CH group of oxazepine ring, ¹³C-NMR spectrum of compounds (9-16) showed signals at (127.1, 129.7, 132.1, 132.9)ppm due to aromatic carbons and signals at (162-167)ppm due to (C=O) of lactone and lactam respectively, signals at (84.2)ppm due to (N-CH) carbon²⁵⁻²⁶.

Scheme 1 : Synthesis of Schiff base and oxazepine R:aromatic aldehyde**5.2 Antimicrobial activity :**

The newly synthesized compounds were screened for their antibacterial activity against *Escherichia coli*, *staphylococcus aureus*, *Salmonella typhii*, *Klebsiella pneumonia*. The results of such studies are given in **Table 5**. The above data showed that compound (12-16) exhibited very good activity against *E.coli*, *S.aureus*, *Salmonella typhii*, *Klebsiella pneumonia*. The remaining compounds were found to have good activity against *E.coli* and *Salmonella typhii* slight or moderate activity against *S.aureus* and *Klebsiella pneumonia*.

5.3. Polarisation measurements :

Table 6 shows the corrosion Potential (E_{corr}), corrosion current (i_{corr}) and Tafel slopes (ba and bc) values of mild steel in 1 mol.L⁻¹H₂SO₄ Solution in the absence and presence of inhibitor of all the eight compounds at 303K calculated from **Scheme(2-9)**. From Table 6 it is clear that compound **10,14,16** offers maximum inhibition efficiency among the eight compounds Schiff base derivatives and the studied compounds suppress the anodic reaction to greater extent than the cathodic one. This behaviour is typical of mixed type inhibitors with anodic predominance.

The difference in the efficiency is referred to the molecular structure effect, which have π -delocalized system of Schiff bases derivatives (C=O) and unshared pairs of electrons of N and O atoms that may cause the increasing or decreasing of the electron density on center of adsorption and leading to an easier electron transfer

from the functional group (C=O-group) to the metal ,producing grater coordinate bonding and hence different adsorption and inhibition efficiency.

Mechanism of corrosion inhibition by Schiff base derivatives:

The transition of metal/solution interface from a state of active dissolution to the passive state is attributed to the adsorption of the inhibitor molecules and the metal surface, forming a protective film. The rate of adsorption is usually rapid and hence, the reactive metal surface is shielded from the aggressive environment, Adsorption process can occur by electrostatic forces between ionic charges or dipoles of the adsorbed species(electrostatic attraction between the positively charged protonated nitrogen atom and negatively charged mild steel surface ,cathodic sites) and the electric charge on the metal surface can be expressed by its potential with respect to the zero charge potential ,Also the inhibitor molecules can be adsorbed species to the vacant electron orbital of low energy in the metal to form a coordinate type of link .Adsorption of inhibitor molecules is often a displacement reaction involving removal of adsorbed water molecules is often adisplacement reaction involving removal of adsorbed water molecules from the metal surface.

Table 1: Melting points, yield, molecular formula (M.F.), molecular weight (M.Wt.) and element analysis of compounds [1-8]

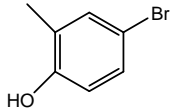
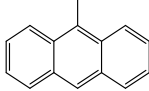
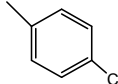
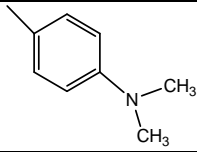
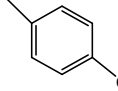
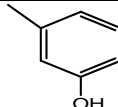
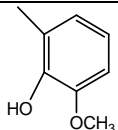
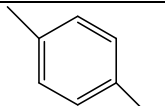
Found			Calculated			M.Wt	M.F.	yield	M.P (^o C)	R	Co mp oun d
C%	H%	N%	C%	H%	N%						
50.89	5.02	9.07	50.90	5.34	9.89	566	C ₂₄ H ₃₀ Br ₂ N ₄ O ₂	70%	107-108		1
83.29	6.95	9.76	83.30	6.99	9.71	576.77	C ₄₀ H ₄₀ N ₄	79%	168-170		2
64.69	6.76	12.46	64.71	6.79	12.56	445	C ₂₄ H ₃₀ Cl ₂ N ₄	52%	105-106		3
72.70	9.13	18.17	72.69	9.15	18.16	462.67	C ₂₈ H ₄₂ N ₆	79%	154		4
77.24	8.96	13.80	77.18	8.97	13.85	404	C ₂₆ H ₃₆ N ₄	80%	102-100		5
70.53	7.69	13.69	70.56	7.90	13.71	408	C ₂₄ H ₃₂ N ₄ O 2	80%	110-111		6
66.60	7.72	11.95	66.64	7.74	11.96	468.59	C ₂₆ H ₃₆ N ₄ O 4	60%	102		7
70.55	7.89	13.69	70.56	7.90	13.71	408	C ₂₄ H ₃₂ N ₄ O 2	79%	93-94		8

Table 2: Melting points, yield, molecular formula (M.F.), molecular weight (M.Wt.) and element analysis of compounds [9-16]

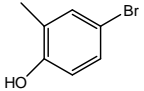
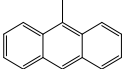
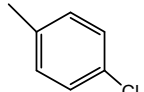
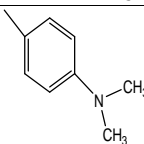
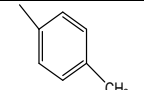
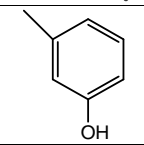
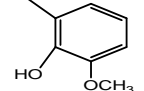
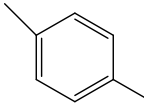
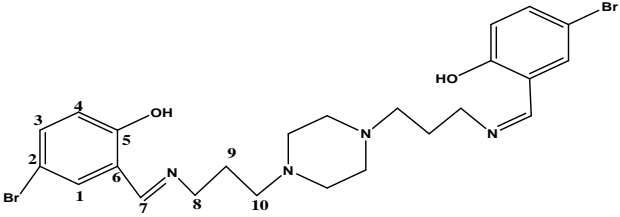
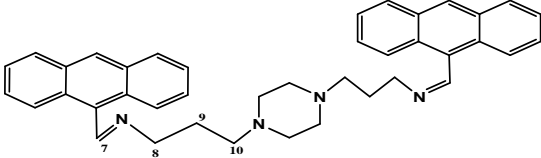
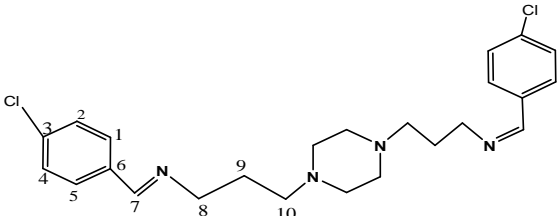
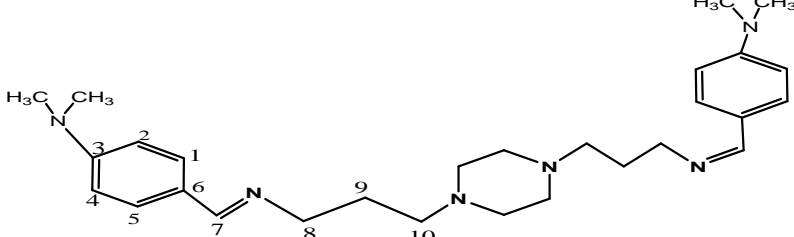
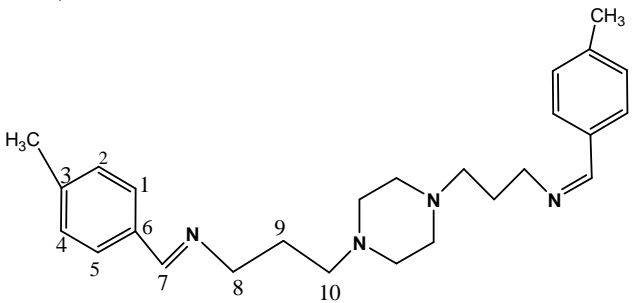
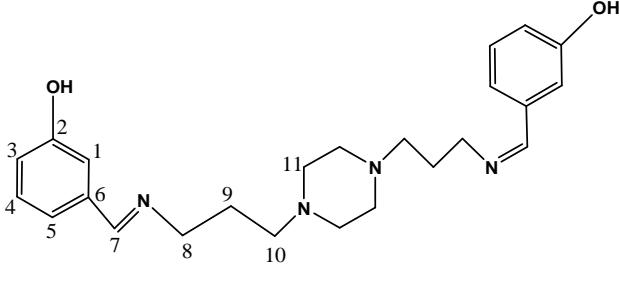
Found			Calculated			M. Wt.	M.F.	Yield (%)	M.P (°C)	R	Compound
C%	H%	N%	C%	H%	N%						
55.69	4.40	6.51	55.70	4.44	6.50	862	C ₄₀ H ₃₈ Br ₂ N ₄ O ₈	70%	164		9
78.29	5.85	6.50	78.30	5.87	6.52	859	C ₅₆ H ₅₀ N ₄ O ₅	69	148-152		10
65.95	5.53	7.69	66.02	5.54	7.70	727	C ₄₀ H ₄₀ Cl ₂ N ₄ O ₅	80	182		11
70.93	7.03	11.27	70.94	7.04	11.28	744	C ₄₄ H ₅₂ N ₆ O ₅	79	186		12
73.40	6.74	8.15	73.45	6.75	8.16	686.84	C ₄₂ H ₄₆ N ₄ O ₅	74	154		13
68.16	5.69	7.94	68.17	5.72	7.95	704	C ₄₀ H ₄₀ N ₄ O ₈	86	147		14
65.95	5.79	7.32	65.96	5.80	7.33	764.82	C ₄₂ H ₄₄ N ₄ O ₁₀	72	168		15
68.13	5.71	7.93	68.17	5.72	7.95	704	C ₄₀ H ₄₀ N ₄ O ₈	73	161		16

Table 3: Spectroscopical data of Synthesized Schiff Base of piperazine

Compound NO	Spectroscopy data
1	<p>IR (Nujol, cm^{-1}): 1635 [(C=N)], 1163(s) [(C-O)].</p> <p>MS (EI): $m/z = 566$.</p> <p>^1H NMR (400 MHz, CDCl_3, ppm) H: 1.81 (m, 4H, 9-H), 2.33-2.40 (m, 12H, 10-H and 11-H), 3.58 ($t^3J=8.0$ Hz), 4H, 8-H), 6.79-7.31 (m, 6H, aromatic ring), 8.20 (s, 2H, 7-H, -C=N), 13.49 (b s, 2H, -OH).</p> <p>^{13}C NMR (400 MHz, CDCl_3, ppm) C: 27.8(C-9), 53.2(C-11), 55.8(C-10), 57.4(C-8), 109.8 (C-6), 119.1(C-5), 120.1 (C-2), 153.2, 154.8 (C-3 or C-4)160.5 (C-5)(aromatic ring), 163.8(C-7, -C=N).</p> 
2	<p>IR (Nujol, cm^{-1}): 1635 [(C=N)], 1163(s) [(C-O)].</p> <p>LC-MS:576</p> <p>^1H NMR (400 MHz, CDCl_3, ppm) H: 1.81 (m, 4H, 9-H), 2.33-2.40 (m, 12H, 10-H and 11-H), 3.58 ($t^3J=8.0$ Hz), 4H, 8-H), 7.39,7.91,8.55 (m, 18H, aromatic ring), 8.12 (s, 2H, 7-H, -CH=N),</p> <p>^{13}C-NMR: (400 MHz, CDCl_3, ppm) : C: 27.8(C-9), 53.2(C-11), 55.8(C-10), 57.4(C-8), 135 (C-6),125.6,128.1,128.9,128.7,131.8,135 (26C,aromatic ring), 160.8(C-7, -C=N).</p> 
3	<p>IR (Nujol, cm^{-1}): 1635 [(C=N)], 1163(s) [(C-O)].</p> <p>LC-MS:445</p> <p>^1H NMR (400 MHz, CDCl_3, ppm) H: 1.80 (m, 4H, 9-H), 2.46 (m, 12H,10-H and 11-H), 3.55 ($t^3J=8.0$ Hz), 4H, 8-H), 7.07-7.31 (m, 8H,aromatic ring), 8.30 (s, 2H, 7-H).</p> <p>^{13}C-NMR: (400 MHz, CDCl_3, ppm):: H:29.8(C-9),52.9(C-11),51.7(C-10),58.1(C-8),118.9(C-6) 116.9, 118.3,128.9,130.6 (C-1,C-2,C-4,C-5) ,136.6(C-3),164.9(C-7,C=N).</p> 
4	<p>IR (Nujol, cm^{-1}): 1636.3 [(C=N)], 2930.31(s) [(C-Haliphatic)].</p> <p>LC-MS:462.67</p> <p>^1H NMR (400 MHz, CDCl_3, ppm) H: 1.80 (m, 4H, 9-H), 2.46 (m, 12H,10-H and 11-H),3.02(s,6H,2(CH_3)N) ,3.55 ($t^3J=8.0$ Hz), 4H, 8-H), 7.07-7.31 (m, 8H,aromatic ring), 8.30 (s, 2H, 7-H).</p> <p>^{13}C-NMR: (400 MHz, CDCl_3, ppm):: H:29.9(C-9),41.2(2CH_3),53.2(C-11),52.7(C-10),59.1(C-8),119.5(C-6) 116.5, 118.2,128.9,131.3(C-1,C-2,C-4,C-5) ,151.9(C-3),164.9(C-7,C=N).</p>

	
5	<p>IR (Nujol, cm^{-1}): 1635 [(C=N)], 1163(s) [(C-O)].</p> <p>LC-MS:404.5</p> <p>^1H NMR (400MHz, CDCl_3, ppm) H: 1.87 (m, 4H, 9-H) ,2.41(s,6H,2CH_3), 2.47 (m, 12H,10-H and 11-H), 3.58 (t(3J=8.0 Hz), 4H, 8-H), 6.79-7.31 (m, 8H,aromatic ring), 8.33 (s, 2H, 7-H)</p> <p>^{13}C NMR (400 MHz, CDCl_3, ppm) 28.8(C-9), 56.1(C-11), 56.7(C-10), 58.3(C-8), 118.9 (C-6),116.9, 118.3, 131.0, 132.0 (C-1,C-2,C-4,C-5), 140.7(C-3)(aromatic ring), 164.5(C-7, -C=N).</p> 
6	<p>IR (Nujol, cm^{-1}): 1634 [(C=N)], 1163(s) [(C-O)].</p> <p>MS (EI): m/z =408</p> <p>LC-MS: 408.5</p> <p>^1H NMR (400 MHz, CDCl_3, ppm) H: 1.87 (m, 4H, 9-H), 2.47 (m, 12H,10-H and 11-H), 3.58 (t(3J=8.0 Hz), 4H, 8-H), 6.79-7.31 (m, 8H,aromatic ring), 8.33 (s, 2H, 7-H), 13.51 (s, 2H, -OH).</p> <p>^{13}C NMR (400MHz, CDCl_3, ppm) 27.8(C-9), 53.1(C-11), 55.7(C-10), 57.3(C-8), 118.8 (C-6),116.9, 118.3, 131.0, 132.0 (C-1,C-2,C-3,C-5), 161.3(C-2)(aromatic ring), 164.9(C-7, -C=N).</p> 
7	<p>IR (Nujol, cm^{-1}): 3300.65 [O-H],1632.45 [(C=N)], 1156.12 [(C-O)].</p> <p>LC-MS :468.59</p> <p>^1H NMR (400 MHz, CDCl_3, ppm) H: 1.87 (m, 4H, 9-H), 2.47 (m, 12H,10-H and 11-H), 3.58 (t(3J=8.0 Hz), 4H, 8-H), 3.83 (s,6H, 2OCH_3), 6.79-7.31 (m, 6H,aromatic ring), 8.13 (s, 2H, 7-H), 9.83 (s, 2H, -2OH).</p> <p>^{13}C NMR (400MHz, CDCl_3, ppm): 27.8(C-9), 53.1(C-11), 55.7(C-10),56.1(-2OCH_3) 57.3(C-8), 118.8 (C-6),116.9, 118.3, 131.0, 132.0 (C-3,C-5,C-4), 150.1(C-1),151.2(C-2),151.5(C-2)(aromatic ring), 165.9(C-7, -C=N).</p>

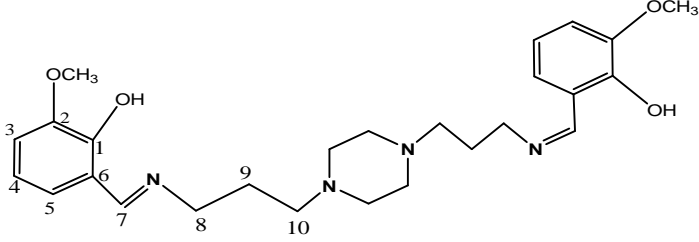
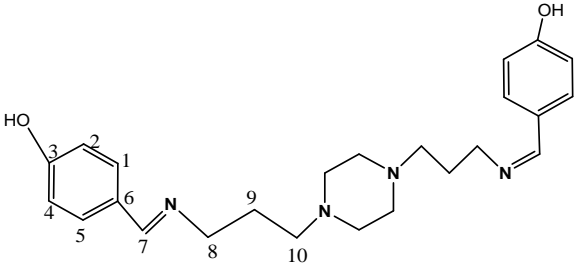
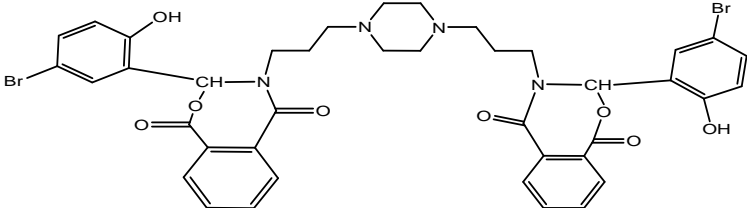
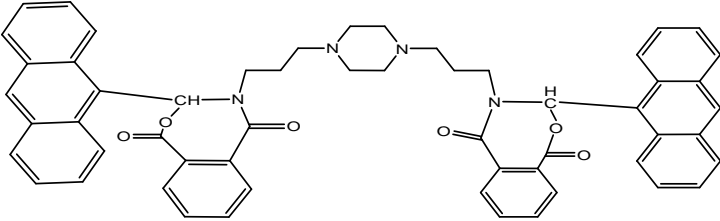
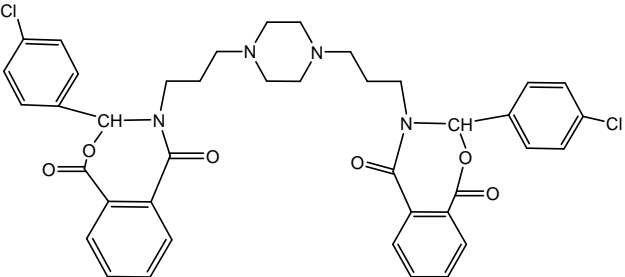
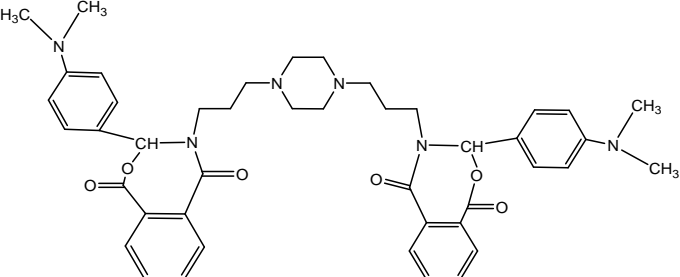
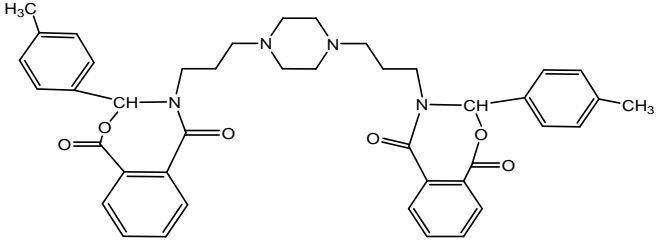
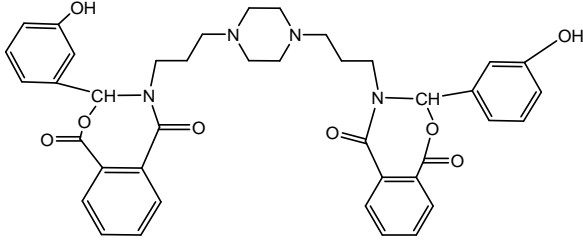
	
8	<p>IR (Nujol, cm^{-1}): 3056.62 (OH), 1644.02 [C=N], 1163(s) [C-O].</p> <p>LC-MS: m/z :408</p> <p>^1H NMR (400MHz, CDCl_3, ppm) H: 1.87 (m, 4H, 9-H), 2.47 (m, 12H, 10-H and 11-H), 3.58 ($t^3J=8.0$ Hz), 4H, 8-H), 6.79-7.31 (m, 8H, aromatic ring), 8.33 (s, 2H, 7-H), 13.51 (b s, 2H, -OH).</p> <p>^{13}C NMR (400 MHz, CDCl_3, ppm) 27.8(C-9), 53.1(C-11), 55.7(C-10), 57.3(C-8), 118.8 (C-6), 116.9, 118.3, 131.0, 132.0 (C-1, C-2, C-3, C-5), 161.3(C-3)(aromatic ring), 164.9(C-7, -C=N).</p> 

Table 4: Spectroscopical data of Synthesized oxazepine compounds

Compound NO	Spectroscopy data
9	<p>IR, (cm^{-1}): :3284.18 [OH], 1709.59 [C=O Lactone], 1634.38 [C=O lactam], 1381.75(O-C-O)</p> <p>MS (EI): m/z =862.56</p> <p>^1H NMR (400 MHz, CDCl_3, ppm) H: 1.81 (m, 4H, 9-H), 2.33-2.40 (m, 12H, 10-H and 11-H), 3.58 ($t^3J=8.0$ Hz), 4H, 8-H), 6.79-7.31 (m, 6H, aromatic ring), 7.35(s, 2H, N-CH), 13.49 (s, 2H, -OH), 7.62-8.15(m, 8H, Aromatic (oxazepine))</p> <p>^{13}C NMR (400 MHz, CDCl_3, ppm) C: 27.8(C-9), 53.2(C-11), 55.8(C-10), 57.4(C-8), 84.2(N-CH) 109.8 (C-6), 119.1(C-5), 120.1 (C-2), 132.9, 132.1, 129.7, 127.1(12C, Aromatic of phthalic anhydride) 153.2, 154.8 (C-3 or C-4) 160.5 (C-5)(aromatic ring), 162, 169.9 (2C=O OF Lactone and lactam respectively)</p> 
10	<p>IR: 3120(C-H arom), 2929.15(C-H aliphatic), 1707.60 [C=O Lactone], 1637.45 [C=O lactam], 1375.55(O-C-O)</p> <p>LC-MS: 873</p> <p>^1H NMR (400 MHz, CDCl_3, ppm) H: 1.81 (m, 4H, 9-H), 2.33-2.40 (m, 12H, 10-H and 11-H), 3.58 ($t^3J=8.0$ Hz), 4H, 8-H), 7.39, 7.91, 8.55 (m, 18H, aromatic ring), 7.35 (s, 2H, -CH=N)</p>

	<p>,7.62,7.78,8.15(m,8H,Aromatic carbons) ¹³C-NMR: (400 MHz, CDCl₃, ppm) :) C: 27.8(C-9), 53.2(C-11), 55.8(C-10), 57.4(C-8), 82.5(N-CH).135 (C-6), 125.6,128.1,128.9, 128.7,131.8,135 (26C,aromatic ring), 161.2,168.5 (2C=O,Of Lactone and lactam respectively),127.1,129.7,132.1,132.9 (12C, Aromatic ring of phthalic anhydride)</p> 
11	<p>IR : 3011.52(C-H arom), 2937.35(C-H aliphatic),1709.60 [C=O Lactone),1647.45 [C=O lactam], 1166.98(Ar-Cl) LC-MS:741.66 ¹H NMR (400 MHz, CDCl₃, ppm) H: 1.80 (m, 4H, 9-H), 2.46 (m, 12H,10-H and 11-H), 3.55 (t(3J=8.0 Hz), 4H, 8-H), 7.35 (s, 2H, 7-H).7.07-7.31 (m, 8H,aromatic ring),7.62,7.78,7.81,8.15)m,4H,aromatic ring of phthalic anhydride) ¹³C-NMR: (400 MHz, CDCl₃, ppm):: H:29.8(C-9),52.9(C-11),51.7(C-10),58.1(C-8), ,84.2(C-7,C=N),118.9(C-6) 116.9, 118.3,128.9,130.6(C-1,C-2,C-4,C-5),136.6(C-3), 127.1,129.7,132.1, 132.9(12C,Aromatic of phthalic anhydride),161.2,167(2C=O, Of Lactone and lactam respectively),</p> 
12	<p>IR: 3015.74(C-H arom), 2985.75(C-H aliphatic),1710.61 [C=O Lactone),1696.49 [C=O lactam], 1581(C-N) LC-MS:759 ¹H NMR (400 MHz, CDCl₃, ppm) H: 1.80 (m, 4H, 9-H), 2.46 (m, 12H,10-H and 11-H),3.02(s,6H,2(CH₃)₂N) ,3.55 (t(³J=8.0 Hz), 4H, 8-H), 7.07-7.31 (m, 8H,aromatic ring), 7.35 (s, 2H, N-CH),7.62,7.78,7.81,8.16(m,4H,Aromatic of phthalic anhydride) ¹³C-NMR: (400 MHz, CDCl₃, ppm):: H:29.9(C-9),41.2(2CH₃),53.2(C-11),52.7(C-10),59.1(C-8),84.2(N-CH) 119.5(C-6) 116.5, 118.2,128.9,131.3(C-1,C-2,C-4,C-5) ,151.9(C-3),127.1,129.7,132.9,132.1 (12C,Aromatic carbones of phthalic anhydride), 161.2,167 (2C,2C=O Of Lactone and lactam respectively).</p> 
13	<p>IR: 3035.74(C-H arom), 2996.75(C-H aliphatic),1718.69 [C=O Lactone),1698.78 [C=O</p>

	<p>lactam],</p> <p>LC-MS:700</p> <p>¹H NMR (400MHz, CDCl₃, ppm) H: 1.87 (m, 4H, 9-H) ,2.41(s,6H,2CH₃) , 2.47 (m, 12H,10-H and 11-H), 3.58 (t(³J=8.0 Hz), 4H, 8-H), 6.79-7.31 (m, 8H,aromatic ring), 7.35 (s, 2H, N-CH) ,7.62,7.78,7.81,8.15(m,8H,aromatic ring of phthalic anhydride)</p> <p>¹³C NMR (400 MHz, CDCl₃, ppm) 28.8(C-9), 56.1(C-11), 56.7(C-10), 58.3(C-8), 118.9 (C-6),116.9, 118.3, 131.0, 132.0 (C-1,C-2,C-4,C-5), 140.7(C-3)(aromatic ring), 84.2(, N-CH),127.1,129.7,132.9(12C,Aromatic ring of phthalic anhydride),161.2,167(2C,2C=O OF Lactone and lactam respectively)</p> 
<p>14</p>	<p>IR:3360(OH),3028(C-H arom),2872(C-H aliphatic), 1715.61 [C=O Lactone),1696.49 [C=O lactam],</p> <p>LC-MS :704.77</p> <p>¹H NMR (400 MHz, CDCl₃, ppm) H: 1.87 (m, 4H, 9-H), 2.47 (m, 12H,10-H and 11-H), 3.58 (t(³J=8.0 Hz), 4H, 8-H), 7.35 (s, 2H, N-CH) 6.79-7.31 (m, 8H,aromatic ring), 7.62,7.78,7.81, 8.45 (m,4H,Aromatic phthalic anhydride) , 13.51 (s, 2H, -OH).</p> <p>¹³C NMR (400MHz, CDCl₃, ppm) 27.8(C-9), 53.1(C-11), 55.7(C-10), 57.3(C-8), 84.5(N-CH),118.8 (C-6),116.9, 118.3, 131.0, 132.0 (C-1,C-2,C-3,C-5),161.3(C-2)(aromatic ring), 127.1,129.7,132.9 (Aromatic carbones of phthalic anhydride) 161.2,167.2(2C,2C=O OF Lactone and lactam respectively)</p> 
<p>15</p>	<p>IR:3371(OH),3056(C-H arom),2950(C-H aliphatic), 1748.93 [C=O Lactone),1698.49 [C=O lactam],</p> <p>LC-MS :764.82</p> <p>¹H NMR (400 MHz, CDCl₃, ppm) H: 1.87 (m, 4H, 9-H), 2.47 (m, 12H,10-H and 11-H), 3.58 (t(³J=8.0 Hz), 4H, 8-H), 3.83 (s,6H, 2OCH₃), 6.79-7.31 (m, 6H,aromatic ring), 7.35 (s, 2H, CH-N), 9.83 (s, 2H, -2OH),7.62,7.78,7.81,8.15(m,4H,aromatic rig of phthalic anhydride)</p> <p>¹³C NMR (400MHz, CDCl₃, ppm): 27.8(C-9), 53.1(C-11), 55.7(C-10),56.1(-2OCH₃) 57.3(C-8), 78.3(N-CH),118.8 (C-6),116.9, 118.3, 131.0, 132.0 (C-3,C-5,C-4), 150.1(C-1),151.2(C-2),151.5(C-2) (aromatic ring), 127.1,129.7,132.1,132.9(Aromatic carbons of phthalic anhydride),161.2,167(2C,2C=O)</p>

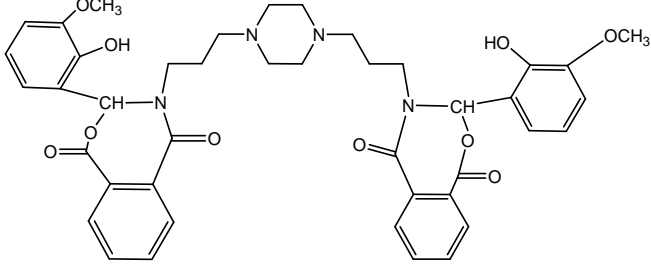
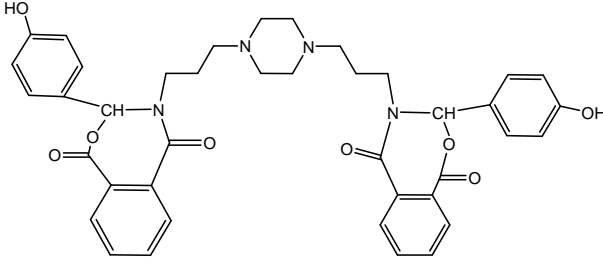
	
16	<p>IR:3360(OH), ,3072(C-H arom),2956(C-H aliphatic), 1749.03 [C=O Lactone),1699.05 [C=O lactam],</p> <p>LC-MS: 704.77</p> <p>¹H NMR (400MHz, CDCl₃, ppm) H: 1.87 (m, 4H, 9-H), 2.47 (m, 12H,10-H and 11-H), 3.58 (t³J=8.0 Hz), 4H, 8-H), 6.79-7.31 (m, 8H,aromatic ring) ,7.35 (s, 2H, <u>CH</u>-N),, 7.62,7.78,7.81,8.15 (m,4H,aromatic rig of phthalic anhydride) , 13.51 (b s, 2H, -OH).</p> <p>¹³C NMR (400 MHz, CDCl₃, ppm) 27.8(C-9), 53.1(C-11), 55.7(C-10), 57.3(C-8),84.2(<u>CH</u>-N) ,118.8 (C-6),116.9, 118.3, 131.0, 132.0 (C-1,C-2,C-3,C-5), 161.3(C-3)(aromatic ring), 127.1, 129.7, 132.1, 132.9(Aromatic carbons of phthalic anhydride), 161.2, 167(2C,2C=O) .</p> 

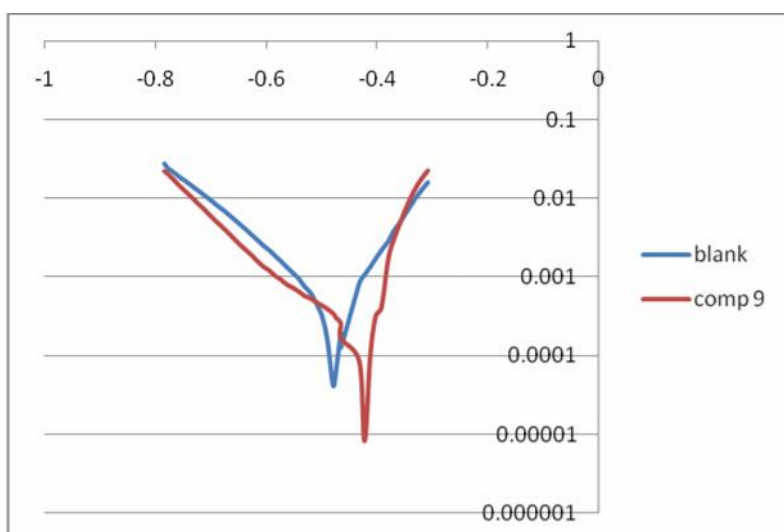
Table 5: Antimicrobial activity for prepared compounds

<i>Klebsiella pneumonia</i>	<i>Sal. typhi</i>	<i>E. coli</i>	<i>Staph. aureus</i>	Comp. No.
++	++	±	±	9
++	++	±	±	10
++	++	±	±	11
++	±	++	±	12
++	±	++	±	13
±	++	++	++	14
±	++	++	++	15
±	++	++	++	16

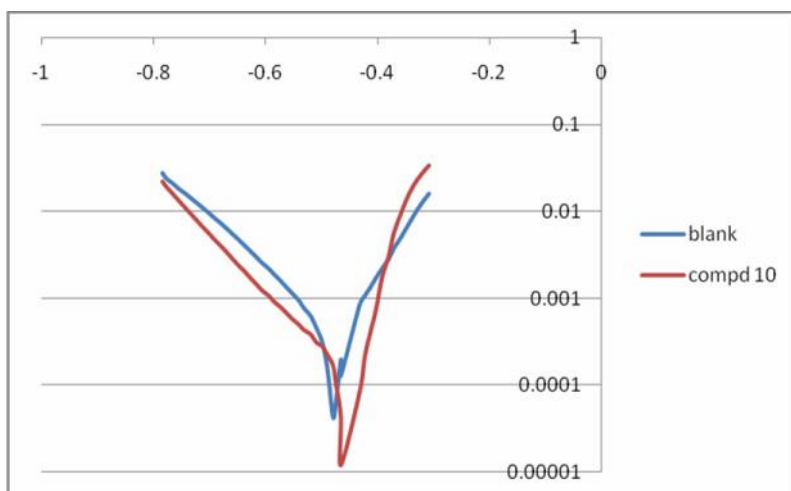
Key the symbols :(-) = No inhibition , (±) = 6-9 mm, (++) = 15-22 mm.

Table 6: Corrosion kinetic parameters of mild steel exposed to 1 mol.L⁻¹H₂SO₄solution in absence and presence of inhibitors

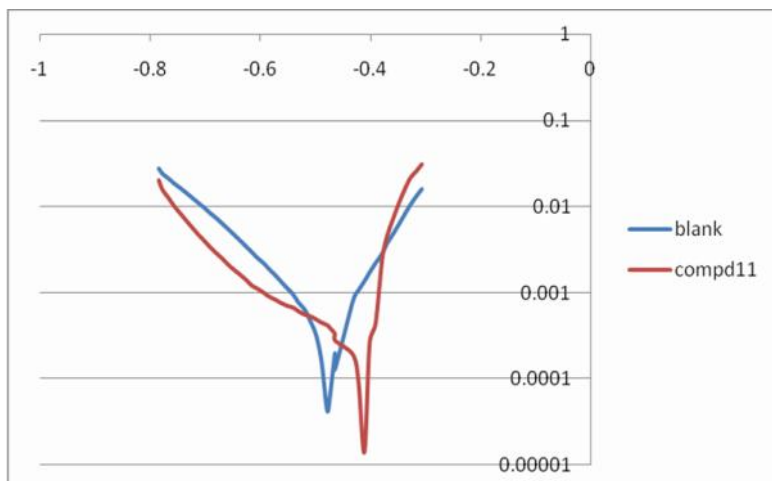
IE(Using icorr) %	Rp	bc / (mv.dec -1)	ba / (mv.dec -1)	icorr/ $\mu\text{A}\cdot\text{cm}^{-2}$	-Ecorr/mv	Comp Name
BLank		12.43	6.38	480	480	blank
62.70	102.95	27.49	5.019	179	400	9
89.58	496.27	14.55	10.09	50	520	10
63.95	95.614	25.78	4.47	173	420	11
63.54	95.95	25.78	4.55	175	418	12
58.54	71.13	19.52	6.28	199	450	13
85.16	330.54	11.17	10.19	70	500	14
79.16	221.58	14.55	7.86	100	510	15
87.5	349.83	11.82	8.18	60	520	16



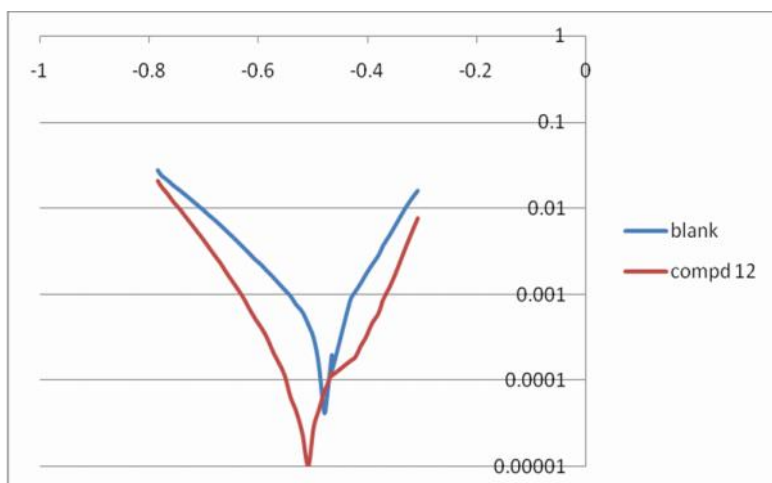
Scheme (2) Tafel plots obtained for mild steel corrosion in absence and presence of compound (9)



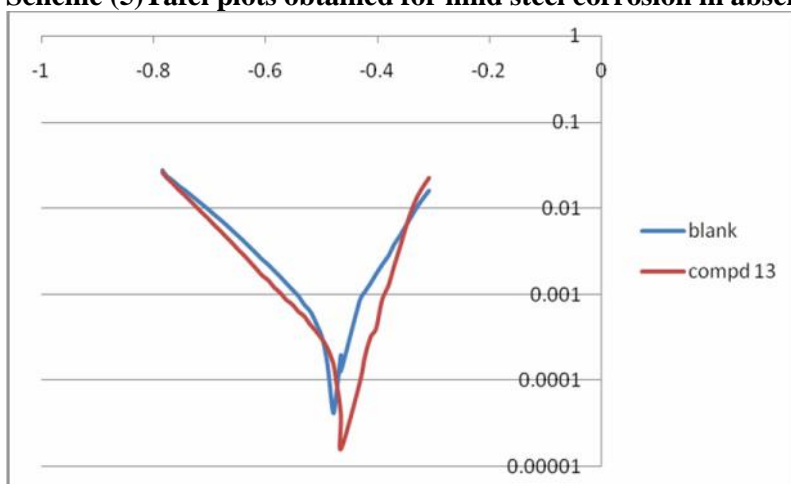
Scheme (3) Tafel plots obtained for mild steel corrosion in absence and presence of compound (10)



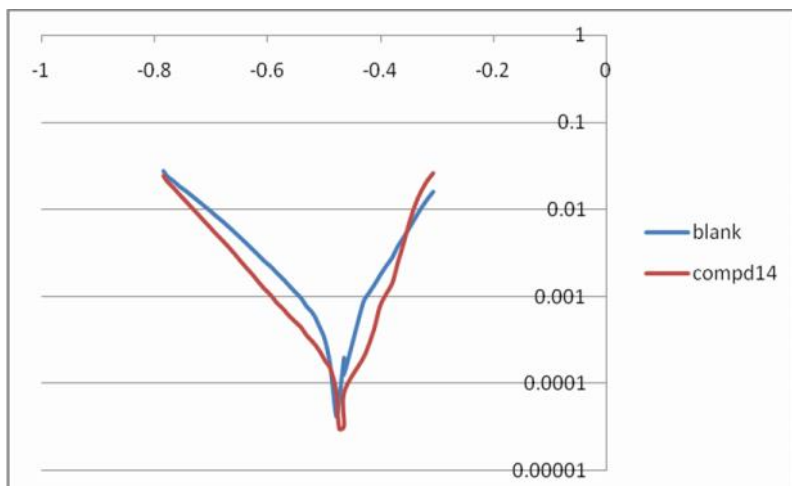
Scheme (4) Tafel plots obtained for mild steel corrosion in absence and presence of compound(11)



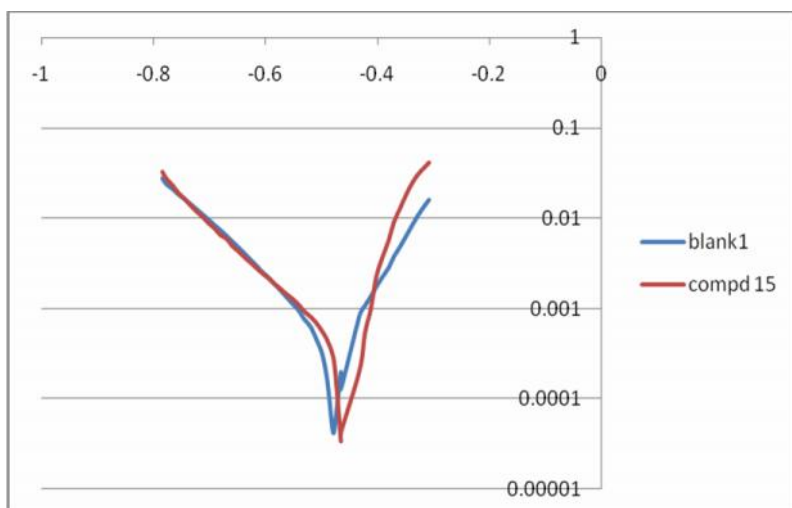
Scheme (5) Tafel plots obtained for mild steel corrosion in absence and presence of compound (12)



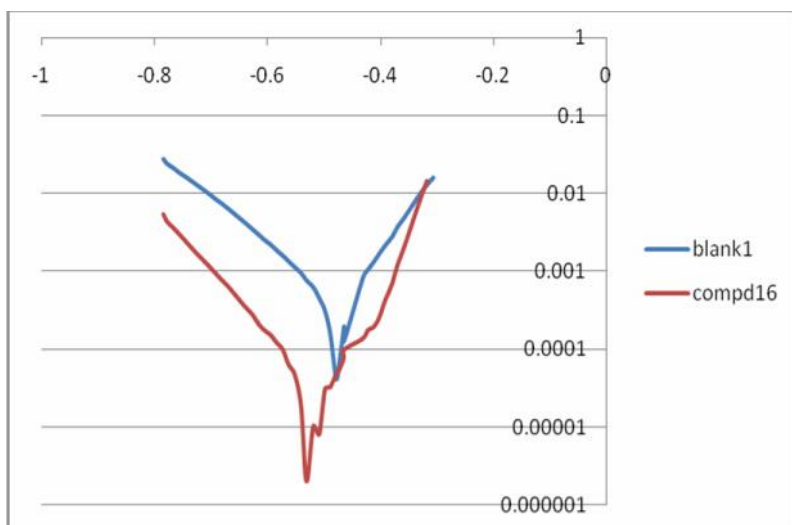
Scheme (6) Tafel plots obtained for mild steel corrosion in absence and presence of compound (13)



Scheme (7) Tafel plots obtained for mild steel corrosion in absence and presence of compound (14)



Scheme (8) Tafel plots obtained for mild steel corrosion in absence and presence of compound (15)



Scheme (9) Tafel plots obtained for mild steel corrosion in absence and presence of compound (16)

6. Conclusions

The main aim of the present study is to synthesize and investigate the antimicrobial and anti corrosion activity of new heterocyclic derivatives containing oxazepine ring with the hope of discovering new structures serving as potential broad spectrum antimicrobial agents and anti corrosion agents ,the antibacterial revealed that nature of substituents on the phenyl ring viz., methyl, methoxy, hydroxyl, dimethylamino, chloro, bromo and hydroxyl group at the para positions of the aryl moieties are determinant for the nature and extent of the anti-bacterial activity of the synthesized compounds, which might have influences on their inhibiting mechanism of actions. Compound (13-16) which contain electron donating functional moiety is most potent against bacterial it's showed good antimicrobial activity. from the results it is obvious that all eight studied compounds function as effective corrosion inhibitors in 1 mol.L⁻¹H₂SO₄ medium with compounds **10, 14,16** being the best of eight compounds.

References

1. Ktritzky A.R.; Vakulenko A.V.; Gedu R. A., Roger J.W, "The Pharmacological Basis of Therapeutic"; ARKIVOCI, 85. pp. : 1153-1158, 2007.
2. Katritzky A.R. Vakulenko A. V; Gedu R. A. and Rogers J. W.; "The new derivative of 3-[(2-morpholine-4-yl)ethyl]-4-substituted-1,2,4-triazoline-5-thiole" ARKIVOCI, 8555 – 60, 2007.
3. Ye. X; Chen. Z. Zhang A.; "Synthesis and Anti-Inflammatory Activity of Chalcone and Related Mannch Bases Medical Chemistry", *Molecules*, 12, 1202 – 10, 2007.
4. Khalaf A. J, "Synthesis and Antibacterial Activities of new Metronidazole and Imidiazole Derivatives", *Molecules*, 14: PP. 2431–2446, 2009.
5. C.O. Wilson and O. Givold, "Text book of Organic Medicinal and pharmaceutical Chemistry", Ptiman Medical Publishing Co.London ccopy right .Cby. J.B. Lippin Cott Company, 5th Edition, 1966
6. H. Matsuzaki, I. Takuchi, Y. Hamad and K. Hatano, *Chem. pharm. Bull*, 48(5) Chem. 755 (2000).
7. Abed M. Daher Al-Joubory. Ph.D thesis University of Tikrit , "Synthesis and Comparative Study of some Heterocyclic Compounds Containing Five and Seven – Membered Rings By Conventional and Microwave Method (MAOS) and Evaluate the Biological activity of Some of Them", Pp. 103–105, (2010).
8. B. Hue, B. Palomba, M. Giacordy-pety-Bottai, R.Alric and p. Prtit, *Ther. Drug Mani.*, 20, 335-339 (1998).
9. S. Kapur, R. Cho, C. Jones, G. Mckay and R. B. Zipursky. *Biol. Psychol.*, 45, 1217-1220 (1999).
10. H. Takeuchi, S. Ykota, S. Shimada, Y. Ottani, S. Miura and H. Kubo, *Curr. Ther. Res. Chin. Exp.* 53, 427-434, (1993)
11. Afaf H. Al-Masry, H.H. Fahmy and S.H. Ali Abdel Wahed, *Molecules*, 5, 1429, 2000.
12. Juburi.R, Synthesis and Characterization of Some Heterocyclic Compounds (Oxazepine,Tetrazole) Derived from Schiff Bases, *Journal of Al-Nahrain University Vol.15 (4), December, 2012, pp.60-67*
13. C.Kustu,K.C.Emregul,O,Atakol,*Corros.Sci.*49(2007)2800.
14. M.Behpour,S.M.Ghoreishi,M.Salavati-Niasari,B.Ebrahimi,*Mater.Chem.Phys.*107(2008)153.
15. M.Bouklah,A.Ouassini,B.Hammouti,A.El Idrissi,*Appl.Surf.Sci.*252(2006)2178.
16. M.Elayyachy,A.El Idrissi,B.Hammouti,*Corros.Sci.*48(2006)2470.
17. Ghosh, R.; Rahaman, S. H.; Lin, C.; Lu, T. B.; Ghosh, K. *Polyhedron* 2006, 25, 3104
18. Al-Juburi, Ruaa, Synthesis and Characterization of Some Heterocyclic Compounds (Oxazepine, Tetrazole) Derived from Schiff Bases, *Journal of Al-Nahrain University, Vol.15 (4), December, 2012, pp.60-67.*
19. AL-JAMALI, MAHMOOD, NAGHAM, Synthesis and Identification of Oxazipen, Diazipene Compounds Via Peri cyclic Reactions, *J. Chem. & Cheml. Sci. Vol.3 (2), 64-69 (2013).*
20. L. Smith, W. C. Wong, A. S. Kiselyov, S. Burdzovic-Wizemann, Y. Mao, Y.Xu, M. A. J. Duncton K.Kim, E.L.Piatnitski, J.F.Doody, Y.Wang, R.L.Rosler, D.Milligan, J.Columbus ,C.Balagtas, S.P.Lee,

- A.Konovalov and Y.R.Hadari, Accepted for Publication 11 July 2006,,*Bioorganic and Medicinal Chemistry Letters*, 2006.
21. C.Kustu,K.C.Emregul,O.Atakol,Corros.Sci.49(2007)2800.
 22. Bentiss,F.;Traisnel,M.;Lagrene,M.Corrosion Science 2000,42,127.
 23. Onuchukwa,A.I.;Trasatti,S.P.;Trasatti,S.Corrosion Science 1994,36,1815.
 24. Haqdar.F, Peikari.M,Abootalebi.O,Poorqasemi.E.Investigating accuracy of the Tafel extrapolation method in HCl solutions ,Corrosion Science 51(2009) 1043-1054.
 25. R.M. Silverstein and G.C. Bassler "Spectrometric identification of organic compounds", Johan and Sons, New Yourk , 4th Edition 1981..
 26. D.H. Williams, and Felming; Translated by Sarkss J.Y.; "Spectroscopic method s in organic chemistry", Baghdad University, 1st Edition 1986.

★★★★★