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# Synthesis,Characterization, Biological Evaluation and anti corrosion activity of some new bis-piperidone Derivatives

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## ABSTRACT

Aseries of piperidine derivatives was synthesis by the reaction between substituted benzaldhyde ,pentanone-3,and ammonium acetate by refluxining for 1-2 hr in ethanol and the derivatives refluxed with (4,4'-Diaminodiphenyl sulphone) yielded bispiperidine ,the structures of the synthesized compounds were confirmed by spectroscopy analysis , The reported compounds were screened for their antibacterial activities against : Staphylococcus aureus, Klebsiella,Escherichia coli,pseudomonas aeruginosa,Salmonilla typhi, Enterobacter. The synthesized piperidone-4 were tested to determine their ability to inhibit corrosion of mild steel in 1mol.$L^{-1}$ $H_2SO_4$ and measured by polarization measurements . Which showed that these compounds act as mixed-type inhibitors. The studies revealed that the nitrogen of Schiff base and sulfur atome ,piperidine moiety ,and the phenyl ring assist largely in corrosion control.

**Keywords:**piperidones-4,4,4'-Diaminodipheylsulphone,antibacterialactivity,anticorrosion, polarization, bispiperidine.

## INTRODUCTION

In recent years,there has been growing interest in the synthesis of bioactive compounds in the field of heterocyclic chemistry among the family of heterocyclic compounds ,the nitrogen containing heterocyclic ,especially piperidones-4 have gained considerable importance presumably because of their varied biological properties such as antiviral antitumor [1] analgesic [2] local anaesthetic [3] antimicrobial, bactericidal, fungicidal,herbicidal,insecticidal,antihistamine,anti-inflammatory,anticancer, and central nervous system (CNS) stimulant and depressant activities [4] piperidine derivatives formed by the reaction between pentanone-3,ammonium acetate and substituted aromatic aldehyde .ethanol acts as solvent medium [5]bispiperidine derivatives which are formed by the reaction with (4,4'-diaminodiphenysulphone ) under reflux .it is known fact that mild steel,the material of choice in many industries ,undergoes corrosion in acide medium .efforts are taken worldwide to control the corrosion .organic compounds containing N,O,S and p as heteroatoms or those containing multiple bonds function as good inhibition [6-12] piperidones have been found to inhibit acid corrodion of mild steel [13.]

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## MATERIAL AND METHODS

Melting points (M.P) of the compounds were determined in open capillary method on jindal melting point apparatus .TLC was performed to assess the reactions and the purity of the products . The instruments used for spectroscopic data are IR :Jasco IR -470, Spectrophotometer.$^{13}C$,$^{1}H$-NMR spectra were recorded at 400 MHz spectrophotometer in $CDCl_3$using TMS (Tetra methyl saline )as an internal standard .Mass spectra were recorded on a VG analytical 7070E instrument equipped with VG 11-250 data acquisition system.

**1. Synthesis :**

**Step I : Synthesis of piperidine derivatives (1a-e):**

Pentanone-3(0.01mol) ,substituted benzaldhyde (0.02 mol) and ammonium acetate (0.01 mol) were taken in a 500ml round bottom flask .further ethanol (25 ml) was added to the flask and mixed well ,so as to make ahomogenous mixture then this mixture was refluxed at 80 0C For 1-3 hr once the reaction was completed the mixture was poured over cooled ice . the crude product obtained was filtered and the solid product was collected and washed with cold water . then Which was dried at room temperature ad recrystallizied with ethanol .

**Step II: Synthesis of Bispiperidine Derivatives (2a-e):**

2,6-diphenyl–piperidone-4 derivative(0.02 mol ) Formed in step I and (4,4'- Diamino diphenyl sulphone) (0.01 mol) was taken in around bottom flask dissolved in ethanol ,one drop of concentrated sulphuric acid was added.The reaction mixture was refluxed for 24h.The reaction mixture was then poured into crushed ice. Separated solid was filtered ,dried ad recrystallized from ethanol and water .The reaction was monitored by TLC .

**2. Polarization measurements :**

Electrochemical measurements

Were carried out in conventional three –electrode system in CHI 604 instrument (USA)at 303 K. The working electrode (mild steel) has ageometric area of 1 $cm^2$.The saturated calmmel and platinum electrodes were used as reference and auxiliary electrodes. Equation (1) show the calculation of IE from corrosion current :

$$IE = \left(1 - \frac{icorri}{icorr0}\right) \times 100 \qquad (1)$$

**3. Pharmacology:**

Antibacterial activities of the tested compounds (2a-2d) were assessed in vitro against each four-representative bacterial species viz ,Staphylococcus aureus[gram-positive],Escherichia coli,Klebsilla Pneumonia ,Salmonella typhi ,Enterobacter[gram-negative],the compounds were tested at aconcentration of 1000 ,500,250 ppm .The zone of inhibition was measured in millimeters.

## RESULT AND DISCUSSION

### 1. Synthesis:

The structures of synthesized compounds were confirmed by TLC,mp,IR,MS and $^{1}H$,$^{13}C$-NMR spectroscopy method.Table 1,2 shows the M.P, Yield of prepared compounds. The synthetic procedure adopted to obtain the target compounds are depicted in Scheme 1.The most characteristic FT-IR bands of compound (1a-1d) appeared at (1702.34$cm^{-1}$) for (C=O) which disappeared in the spectrum of compound (2a-2d) with new bands appeared at (1626.36 $cm^{-1}$ ) due to (C=N) and band at (1269.29 $cm^{-1}$) due to (asymmetric–$SO_2$- Stretching) and (1152.94 $cm^{-1}$ ) due to (symmetric–$SO_2$- Stretching). The mass spectra of the synthesized compounds showed the parent peak confirming the molecular weight of the compounds. $^{1}H$-NMR spectrum of compound (2a-2d) showed deplete peaks at δ: 6.66 PPm due to aromatic protons of sulphonyl rings and multiple peaks at(δ :7.18,7.73,7.85 ppm,) Due to aromatic protons of substituted benzene aromatic of piperidine and deplete peaks at 7.95 ppm due to aromatic protons of ( sulphonyl ) , compound (2d) Show single peak at δ: 5.03 ppm Due to O-$\underline{CH_2}$-Ar,compound (2C) Show single peak at δ: 3.80 ppm due to (2O-$\underline{CH_3}$),all compounds show signals at (1.26 ppm) due to ($\underline{CH_3}$-CH) , signals at (1.64 ppm) due to (NH) ,Signals at (2.74 ppm) due to ($\underline{CH}$-$CH_3$)' signals at (4.16 PPm) due to ($\underline{CH}$-NH),in addition to signals at (7.73,7.85 )due to protons of aromatic ring of piperidons, $^{13}C$-NMR spectrum of compounds (2a-2d) show no signals in the C=O range And show signal at (188.42 ppm ) due to (C=N),signals at (125.80, 129.64, 136.62, 142.16PPm ) due to carbone of sulphonyl aromatic rings ,compound (2a) show signal at(21.10 ppm ) due to (4$CH_3$-Ar) ,compound (2C) show signal at (55.33, 55.54) due to(4$\underline{CH_3}$-O-),) compound (2d) show signal at(70.30 ppm) due to (1C,O-$\underline{CH_2}$-Ar ),All compounds show signal at (10.90 PPm) due to (4$\underline{CH_3}$-CH),signal at (52.17 ppm) due to($\underline{CH}$-$CH_3$), signal at (69.90 ppm) due to (C-2,C-6) ,in addition to signals at (112.19,114.36,128.70,129.33,129.80,131.6) due to aromatic carbons [14,15,16]

### 2. Polarisation measurements :

Table 4 shows the corrosion Potential (Ecorr), corrosion current (I corr) and Tafel slopes (ba and bc) values of mild steel in 1 mol.$L^{-1}$ $H_2SO_4$ Solution in the absence and presence of inhibitor of all the four compounds at 303K calculated from Fig (2a-2d)

### 3. Antibacterial activity :

All the synthesized bispiperidine(2a-2d) exerted potent antibacterial activity in vitro against the tested gram-positive and gram –negative bacterial strains. Table (5), The inhibition zone measured in mm showed that Compound (2C) Exerted strong antibacterial activities against S.aureus , Klebsiella, E.coli, Salmonella, Enterobacter ,while compound (2d)have shown good activity against (S.aureus,Klebsiella ,E.coli) and didn't show any activity against(Salmonella ,Enterobacter), compound (2a) show good activity against

((S.aureus,Klebsiella ,E.coli)) at (1000 ppm) and didn't show any activity against(Salmonella ,Enterobacter )

,compound (2b) show good activity against (S.aureus,Klebsiella ,E.coli)) and mild activity against Enterobacter and Salmonella.

## 4. Conclusion

Several substituted piperidone and its derivatives were synthesized .compounds 1a-1d were prepared ,starting with substituted benzaldhyde, Pentanone-3 ketone ,and ammonium acetate ,via the Mannich reaction, compound 2a-2d were synthesized via condensation with (4,4'-Diamino diphenyl sulphone). The prepared compounds showed promising antibacterial activity against Gram-positive bacteria S.aureus and Gram – negative bacteria Klebsiella , E.coli, Salmonella, Enterobacter. Results of this study show that the nature of substituents on the phenyl ring viz ,methyl,methoxy,chloro,benzyloxy functions at para positions on 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 ,the presence of electron donating methoxy function moiety in compound C2 is most potent against Staphylococcus,Klebsiella,E.coli,Salmonella,Enterobacter. From the results it is obvious that all the four studied compound function as effective corrosion inhibitors in $1mol.L^{-1}$ $H_2SO_4$medium with compound (C2) being the best of the four then compound (A2) ,compound (B2) and compound (D2),the mechanism of the inhibition processes of the corrosion inhibitors under consideration is mainly the adsorption one.The inhibition efficiency values of examined Schiff bases at acommon concentration of 1 Mm follow the order :C2>A2>B2>D2 .The difference in the efficiency is referred to the molecular structure effect ,to rigidity of π-delocalized system of Schiff bases that may cause the increasing or decreasing of the electron density on center of adsorption and leading to an easier electron transfer from the function group (C=N-group) to the metal ,producing greater coordinate bonding and hence different adsorption and inhibition efficiency,the surface coordination is through the sulfur and the nitrogen atoms attached to the hetero ring ti was concluded that the mode of adsorption depends on the affinity of the metal towards the π –electron clouds of the ring system [17].

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**Table 1 :** physicochemical data of piperidine derivatives

| COM NO | R | Molecular formula | Mol Wt | MP ($^0$C) | Yield (%) |
|---|---|---|---|---|---|
| 1a | $CH_3$ | $C_{21}H_{25}NO$ | 307 | 119 | 70% |
| 1b | Cl | $C_{19}H_{19}Cl_2NO$ | 348 | 109 | 80% |
| 1c | $OCH_3$ | $C_{21}H_{25}NO_3$ | 339 | 150 | 96% |
| 1d | O-$CH_2$-Ar | $C_{33}H_{33}NO_3$ | 491 | 76 | 86% |

**Table2:** Physicochemical data of bispiperidine derivatives

| COM NO | R | Molecular formula | Mol Wt | MP ($^0$C) | Yield (%) |
|---|---|---|---|---|---|
| 2a | $CH_3$ | $C_{54}H_{58}N_4O_2S$ | 827 | 162 | 78% |
| 2b | Cl | $C_{50}H_{46}N_4O_2SCl_4$ | 908 | 150 | 69% |
| 2c | $OCH_3$ | $C_{54}H_{58}N_4O_6S$ | 891 | 109 | 80% |
| 2d | O-$CH_2$-Ar | $C_{78}H_{74}N_4O_6S$ | 1195.51 | 230 | 79% |

**Table 3:** Spectroscopial data of bispiperidine derivatives

| **Compound NO** | **Spectroscopy data** |
|---|---|
| **2a** | **IR** : 3480.21 $cm^{-1}$(N-H), 3309.37$cm^{-1}$(N-H), 2979.43 $cm^{-1}$ (C-H alkanes stretch) ,1697. 95 $cm^{-1}$ ( C=N Imin),1486.10 -1588.70 $cm^{-1}$(Ar C=C stretch),1201.96 $cm^{-1}$ (Asymmetric $–SO_2$-) ,1066.65 $cm^{-1}$ (Symmetric $–SO_2$-),1008.67$cm^{-1}$, 815.57 $cm^{-1}$(=CH Aromatic OOP bending1,4-disubstituted),560.95(-$SO_2$-Scissoring).<br>**MS**:827(1.23%),796(7.79%),743(11.68%),727(27.27%),679(22.07 %),635(19.485),591(22.07%),547(23.37%),703(25.97%) 482(100%),424(62.33%),383(10.38%)308(28.57%),288(5.19%)<br>**$^1$H-NMR($CDCl_3$)(ppm):**0.844(d,4$CH_3$,12H,$^2$J=6.587HZ) ,1.84(S,2NH,2H),2.39(S,4$CH_3$,12H),2.78(m,4H,4CH-$CH_3$ ,$^3J_{CHCH3}$=6.76HZ,$^3J_{CH(2)CH(3)}$=6.58HZ),3.57(d,4H,4CH-NH ,$^3J_{CH(2)CH(3)}$=10.06HZ),6.64,6.66 (d,4H,Ar(Dapsone,$^3$J=8.96HZ)), 7.13(d,8H,Ar(piperidone,$^3$J=7.686HZ,)),7.33,7.35(d,8H,Ar (piperidone$^3$J=7. 13HZ)) ,7.67,7.69 (d,8H,Ar (Dapsone)$^3$J=8.96HZ)<br>**$^{13}$C-NMR : ($CDCl_3$) (ppm):**10.20(4C,4$CH_3$-CH ),21.10 (4C, 4$CH_3$-Ar),52.048(4C,2C-3,2C-5),68.62(4C,2C-2,2C-6) ,114.12,126.60,127.57,128.97,129.11,129.22,131.28 ,137.50,139.16,139.43 ,150.41 (36C,Ar),188.43 (2C,2C=N). |
| **2b** | **IR:**2980.67 $cm^{-1}$($CH_2$ Asymmetric) ,1649.97 $cm^{-1}$(C=N),1587.10 $cm^{-1}$(C=C Stretching (phenyl)),1487.77 $cm^{-1}$(C-CStretching ),1405.75 $cm^{-1}$,1322.82 $cm^{-1}$(Asymetric stretching $SO_2$),1188.3 $cm^{-1}$ symetric stretching $SO_2$ , 1091.66 $cm^{-1}$,980.12 $cm^{-1}$,824.45 $cm^{-1}$(=C-H Ar ) Bend oop ),535.87 $cm^{-1}$(C-Cl Stretching<br><br>**MS:**122(0.519%),163(0.649%),186(0.77%),229(1.29%),288(2.59%) ,323(3.89%),349(5.19%),399(12.98%),424(72.72%),478(31.16%),4 82(100%),531(29.87%),591(28.57%),619(22.07%),664(19.48%),67 9(32.46%),726(31.16%),767(15.58%),795(16.88%),852(9.09%)<br>**$^1$H-NMR($CDCl_3$)**:1.27,1.613ppm(m,8H,4$CH_2$-C=N),3.006ppm(s,2H,2NH), 3.76,4.03(t,4H,4CH-NH),6.98(d,4H,Ar–sulfonyl,$^3$J=16HZ),7.32(d,8H,8CH,Ar, $^3J_{CH(2)CH(3}$=8.41Hz,7.43(d,8H,8CH,$^3J_{(H5(H6)}$=8.23Hz),7.51(4H,Ar–sulfonyl,$^3$J =14.63Hz)<br>**$^{13}$C-NMR ($CDCl_3$)**:37.39(4C,2C-3,2C-5) ,62.29(4C,2C-2,2C-6) ,125.80(4C,4Ar diamino diphenylsulphone) 128.65( 2C-2',2C-6',2C-2",2C-6"), 129.39(2C-3',2C-5',2C-3",2C-5") ,129.64(4C-Ar diaminodiphenylsulphone)),131.15(2C-4',4C-4"),133.30(2C-1',2C-1"),136.62(2C,Ar (diamino diphenyl sulphone),142.16 (2C,Ar(diaminodiphenysulphone ),188.438(2C,2C-4,C=N) |
| **2C** | **IR :**3466.33 $cm^{-1}$,3369.78$cm^{-1}$(NH),2953.76 $cm^{-1}$(C-H stretch ),1625. 45 $cm^{-1}$ (C=N),1590.75 $cm^{-1}$ ,1422.67$cm^{-1}$(Ar C=C Stretch),1252.12$cm^{-1}$(asymmetric $–SO_2$-Stretch ),1165.09 $cm^{-1}$ (Symmetric $–SO_2$-Stretch) ,850.60 $cm^{-1}$ ( =C-H Ar 1,4-disubstituted) |

| | |
|---|---|
| | **MS**:77(7.69%,)122($C_8H_{10}O$,2.56%),147(19.23%), 187(12.82%),236(67.94%),293(100%,$C_{15}H_{20}N_2O_2S$),329(79.48%) ,329(79.48%),393(4230%).,395(15.38%),536(7.69%,$C_{33}H_{33}N_3O_2S$) ,587(14.10%),619(10.25%),663(6.41%)737(1.282%),800(1.28%),, 858(2.56%) ,891(3.84%).<br>**$^1$H-NMR($CDCl_3$):**1.26(12H,d,4$CH_3$-CH) ,1.64(2H,S,2NH),2.74(4H,m,4CH-$CH_3$),3.80(s,6H,2O-$CH_3$),3.83( s,6H,2O-$CH_3$),4.16(d,4H,4CH-NH),6.66(d,4H,4CH-Ar(sulfonyl)),7.18(d,8CH(Ar),2H(3'),2H(3"),2H(5'),2H(5"), 7.73,7.85(,d,8CH-Ar ,2H (2'),2H(2"),2H(6'),2H(6") ,8.30(d,4H,4CH-Ar (sulfonyl))<br>**$^{13}$C-NMR($CDCl_3$):**10.56(4C,4$CH_3$-CH),29.77(4C,4CH-$CH_3$),55.33, 55.54(4C,4$CH_3$-O-),68.26(4C,4CH-NH),114.25,114.36(8C,2C-3',2C-5',2C-3",2C-5"),128.49(C-9,C-13),128.70(C-15,C-19),128.75(2C-2',2C-2"),128.93(2C-6',2C-6")129.33(C-10,C-12) ,129.80(C-10,C-18),131.06(2C-1',2C-1"),134.43(2C,C-11,C-17),156.34(2C,C-8,C-14),159.24(4C,2C-4',2C-4") .188.438(2C,C-4) |
| **2d** | **IR** :2980.25 $cm^{-1}$($CH_2$ asymmetric Stretch),2891.72 $cm^{-1}$($CH_2$symmetric Stretch ),1620.23 $cm^{-1}$(C=N ) 1597.42 $cm^{-1}$(C=C Ar stretch),),1485.17$cm^{-1}$(C-CStretching(phenyl)),1451.84$cm^{-1}$ ($CH_2$Scissoring),1378.31$cm^{-1}$($CH_3$Symmetricdeformation), 1238.69 $cm^{-1}$(Asymmetric –$SO_2$-) ,1144.00 $cm^{-1}$(symmetric –$SO_2$),1104.03 $cm^{-1}$ ,751.22 $cm^{-1}$(=CH Aromatic OOP),549.51 $cm^{-1}$ (-$SO_2$-Scissoring )<br>**MS**:1218.51$[M+Na]^+$(10.63%),1108(8.51%),798(12.76%),627(23.40%),514(29.78%),487(42.55%),339(76.59%),198(55.31%), 147(1000%)<br>**$^1$H-NMR($CDCl_3$)ppm:** 0.893(d,4$CH_3$,12H),2.96,3.23,3.27 (m,4CH-$CH_3$,4H),4.20(m,4CH-NH,4H),5.03(s,4Ar-$CH_2$-O,8H) ,6.63(d,4H,Ar(sulfonyl)),6.93 (m,8H,Ar), 7.19 (m,8H,Ar),7.37 (s,20H,Ar),7.64 (d,4H,Ar-sulfonyl)<br>**$^{13}$C-NMR:**($CDCl_3$):10.90(4C,4$CH_3$-$CH_2$-),52.17(4C,4CH-$CH_3$(2C-3,2C-5)),69.91 (4C,2C-2,2C-6) ,70.30(4C,4O-$CH_2$-Ar) ,112.19 ,114.17 ,121.32 ,126.93 ,127.14 ,127.43, 127.87, 128.08, 128.61,128.71,129.31,131.20 (50C,Ar), 136.94(2C,2C-$SO_2$-C),150.50(2C,Ar (sulfonyl),156.23 (4C,4C-O-$CH_2$-Ar),188.42 (2C,2C=N) |

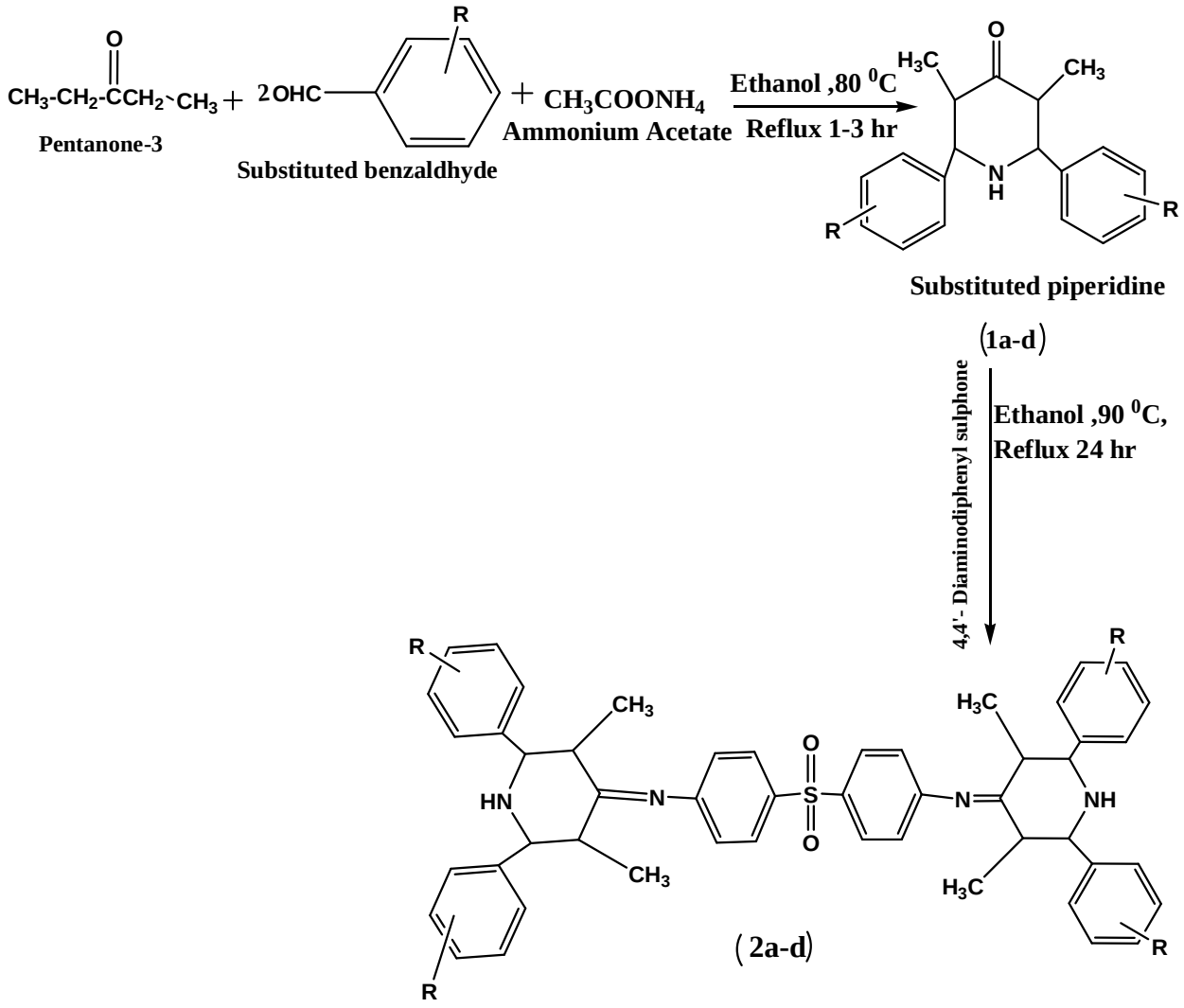
**Table 4 :** Corrosion kinetic parameters of mild steel exposed to 1 mol.$L^{-1}$ $H_2SO_4$ solution in absence and presence of inhibitors

| COMPOUND Name | Rp | bc /(mv.dec -1) | ba /(mv.dec -1) | icorr/ $\mu A.cm^{1-}$ | - Ecorr/mv | IE(Using icorr) % |
|---|---|---|---|---|---|---|
| **BLank** | | **12.43** | **6.38** | **480** | **480** | |
| **2A** | **349.83** | **11.82** | **8.18** | **60** | **520** | **87.5** |
| **2B** | **330.54** | **11.17** | **10.19** | **70** | **500** | **85.16** |
| **2C** | **496.27** | **14.55** | **10.09** | **50** | **520** | **89.58** |
| **2 D** | **116.019** | **18.65** | **7.49** | **110** | **500** | **77.08** |

**Table 5:** Antibacterial activity of prepared compounds Zone of inhibition (in mm)

| Compound | Compound 2a | | | Compound 2b | | | Compound 2C | | | Compound 2d | | | DMSO |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Bacterial | 250 ppm | 500 ppm | 1000 ppm | 250 ppm | 500 ppm | 1000 ppm | 250 ppm | 500 ppm | 1000 Ppm | 250 ppm | 500 ppm | 1000 Ppm | 0 |
| Staphy lococcus | 7 | 10 | 20 | 10 | 10 | 20 | 10 | 20 | 20 | 7 | 7 | 10 | 0 |
| Klebsiella | - | 7 | 20 | 20 | 20 | 20 | 10 | 10 | 30 | 7 | 7 | 10 | 0 |
| E.coli | 7 | 10 | 20 | 7 | 20 | 20 | 7 | 7 | 10 | 7 | 10 | 30 | 0 |
| Salmonella | - | - | - | - | - | 7 | 10 | 20 | 30 | - | - | - | 0 |
| Enterobacter | - | - | - | 7 | 7 | 7 | 10 | 20 | 20 | - | - | - | 0 |

**Figure 1:** Synthetic Schemes for synthesis of bispiperidine derivatives



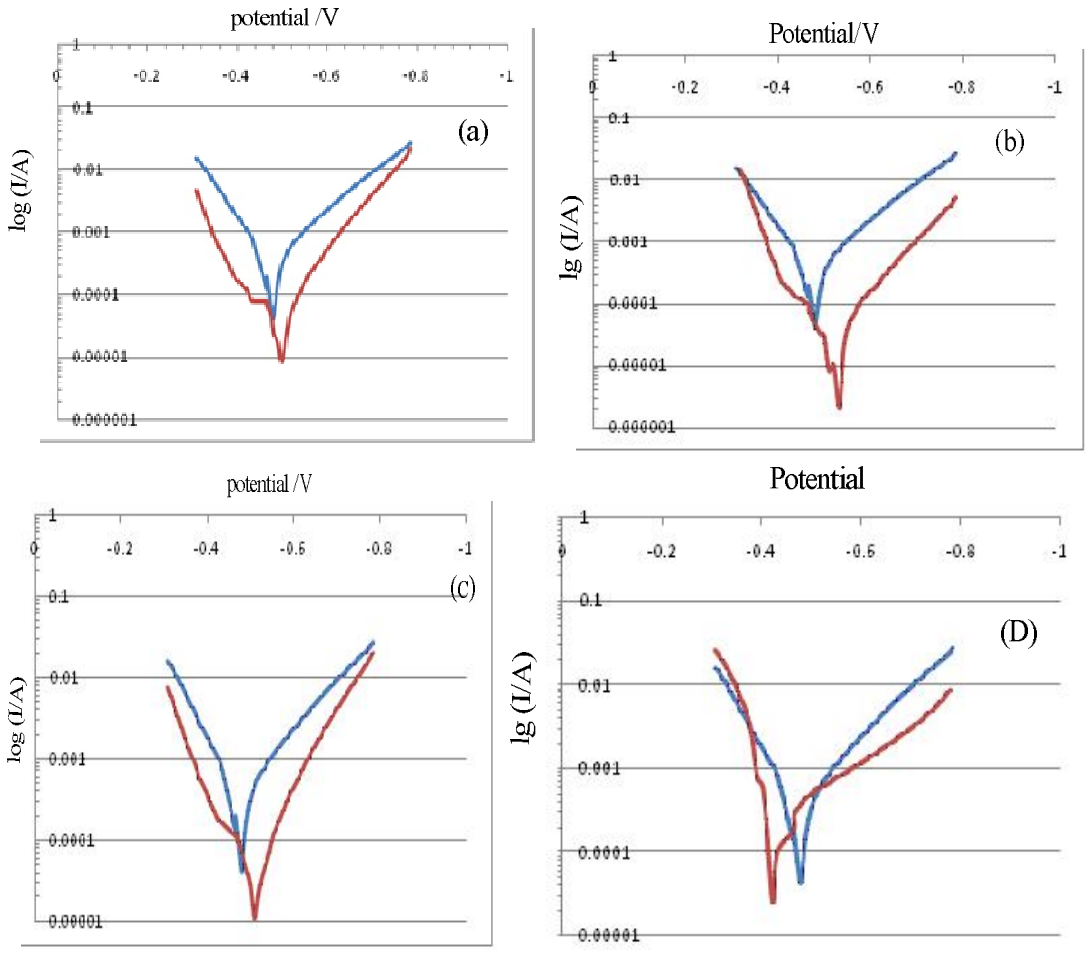



**Fig.2** Tafel plots obtained for mild steel corrosion in absence and presence of (a) compound (2A),(b) compound (2B),(C) compound (2 C), (D) compound (2D)

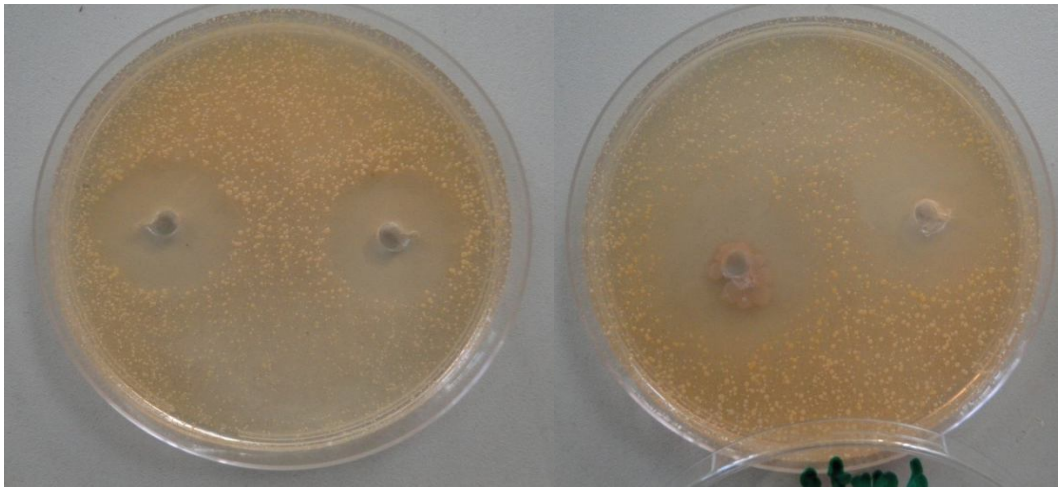


**Fig .3** antibacterial activities of synthesized compounds 2b,2c against Staphylococcus