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# ONE POT SYNTHESIS OF 4-(BENZYLIDENE SUBSTITUTED BENZYLIDENE)-N-ARYL AMINO-2-(STYRYL/SUBSTITUTED STYRYL) 1H-IMIDAZOLE-5(4H)-ONE DERIVATIVES AND THEIR ANTI-BACTERIAL ACTIVITY EVALUATION

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**Abstract**

Synthesis of 4-(benzylidene/substituted benzylidene)-N-aryl amino-2-(styryl/substituted styryl)-1H-imidazole-5(4H)-one derivatives have been carried out with good yields from 4-(benzylidene/substituted benzylidene)-2-methyl- oxazolin-5-ones in presence of phenyl hydrazine, schiff's bases and triethylamine as catalyst and their antibacterial activity against *Escherichia coli, Providencia aeruginosa*, *Pseudomonas azotogensis* and *Baccilus Subtillis* has been carried out by comparing with standard drug *streptomycin*. Some of the synthesized compounds possess good activity against *Escherichia coli* and *Baccilus Subtillis*.

**Keywords:** Green chemistry, Schiff bases and anti-bacterial activity.

## Introduction

The increasing resistance of human pathogens to the current antimicrobial agents is a serious medical problem. Many of the drugs currently available possess undesirable side effects and might be toxic. Considering the fact that the available antimicrobial agent originate from a limited number of sources and that most of them have similar modes of activity, it is very

important to explore additional sources for substances with potential antimicrobial activity, which could possibly have different modes of activity or affect different sites in the bacterial and fungal cells. Imidazole and its derivative are of great significance due to their important roles in biological system. The synthesis of newer class of anti-bacterial and anti-fungal agents is in need of time, especially against drug-resistant bacteria and fungi, such as gram-positive and gram-negative strains, which are responsible for a number of serious infections in the acute and chronic care units in hospitals.

Imidazolinone ring system is of biological and chemical interest since long. The Imidazolinone units are found in many biologically active compounds. The Imidazolones compounds having diverse bioactivities including anticancer[i], anti-HIV agents[ii], anticonvulsant[iii,iv], monoaminooxidase (MAO) inhibitory[v], antiparkinsonian[vi,vii], CNS depression[viii], antimicrobial[ix-xii], anthelmintics [xiii] etc

Schiff's bases in heterocyclisation can act as hydrogen acceptors. Proton acceptor-donor catalyst L-Triethylamine has been playing a vital role in synthetic organic chemistry[xiv]. Amino acids have emerged as an efficient and important catalysts in several transformations such as aldol reactions, conjugate addition[xv], additions to imines and nitro-alkenes[xvi].

This prompted us to synthesize 4-(benzylidene/substituted benzylidene)-N-aryl amino-2-(styryl/substituted styryl)-1H-imidazole-5(4H)-one derivatives from oxazoline-5-one derivatives by making use of schiff's bases and triethylamine as a catalyst and evaluate them for their anti-bacterial activity.

**Experimental:**

Melting points are uncorrected and taken in open capillary tubes in sulphuric acid bath. TLC was run on silica gel – G and visualization was done using UV light. IR spectra were recorded using Perkin – Elmer 1000 instrument in KBr pellets. $^1$H NMR spectra were recorded in $CDCl_3$ using TMS as internal standard with 400 MHZ spectrometer. Mass spectra were recorded on Agilent-LCMS instrument under CI conditions and given by Q+1 value only.

**Preparation of (Z)-2-acetamido-N-phenyl-3-(phenyl/substituted phenyl) prop-2-enamides 2(a-l)**

A mixture of 4-(benzylidene/substituted benzylidene)-2-methyl oxazolin -5-ones **1(a-l)** (10 mM) and phenylhydrazine (10 mM) was dissolved in ethanol and refluxed for 5 h at 80$^o$C. The completion of the reaction was monitored by TLC (1:3 of EtOAc: Hexane) then this

reaction mixture was cooled to room temperature and poured into ice-cold water (50 ml). Solid separated out which was collected, washed with water (10 ml) and dried. The product was recrystallised from ethanol to obtain (Z)-2-acetamido-N-phenyl-3-(phenyl/substituted phenyl)prop-2-enamides **2(a-l).** The formation of **2(a-l)** from 2-oxazolin-5-one derivatives **1(a-l)** has been confirmed from the spectral data.

The IR spectrum of the enamides **2(a-l)** showed peacks for NH group and C=O and absence of the peak for lactone ring. The $^{1}$HNMR spectra of the enamides showed signals for NHNH,NHPh,and NHCO groups. The mass spectrum of the compounds have given molecular ion peacks (M+) corresponding to their molecular weights.

**Preparation of (4Z)-4-benzylidene-1-phenyl-2-styryl-1H-imidazol-5(4H)-one derivatives: 6(a-l)**

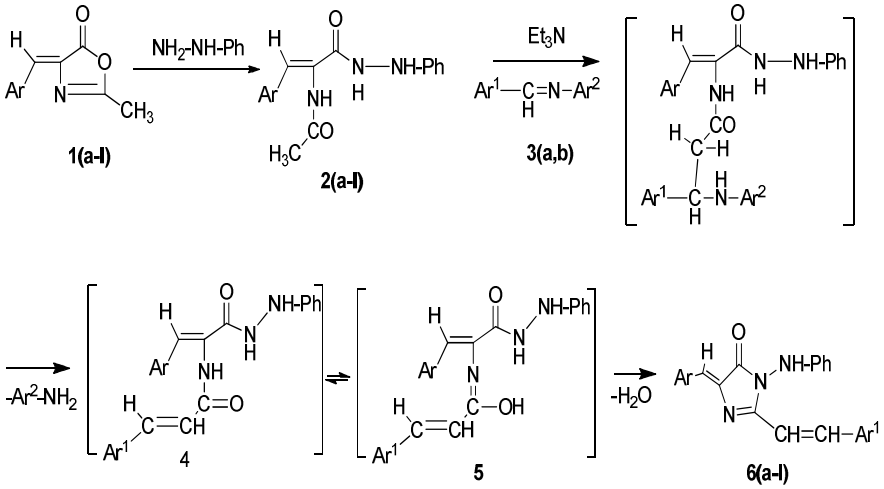
Equimolar quantities of (Z)-2-acetamido-N-phenyl-3-(phenyl/substituted phenyl)prop-2-enamides **2(a-l)** (10mM) and schiff's bases **3(a,b)** (10mM) were mixed together in 20 ml of ethanol in the presence of triethylamine(1 mM) as catalyst. The mixture was refluxed for 2 hrs. The completion of the reaction was monitored by TLC (1:3 of EtOAc: Hexane), then this reaction mixture was cooled to room temperature and poured into ice-cold water (50 ml). The solid separated out was collected, washed with water (10 ml) and dried. The product was recrystallised from ethanol to obtain 4-(benzylidene/substituted benzylidene)-N-aryl amino-2-(styryl/substituted styryl)-1H-imidazole-5(4H)-one derivatives **6(a-l)**.The physical and spectral analysis of the compounds is given below

**Results and Discussion**

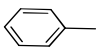
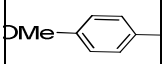
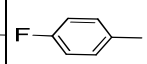
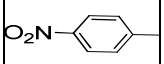
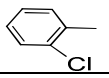
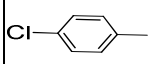
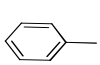
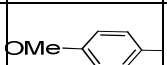
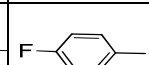
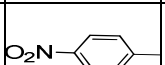
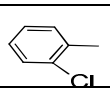
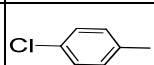
Title compounds 4-(benzylidene/substituted benzylidene)-N-aryl amino-2-(styryl/substituted styryl)-1H-imidazole-5(4H)-one derivatives **6(a-l)** have been synthesized by green approach through synthetic sequence shown in **scheme-I**. Initially, (Z)-4-(benzylidene/substituted benzylidene)-2-methyl oxazol-5(4H)-ones 1**(a-l)** were treated with phenylhydrazine and refluxed for 5 h in ethanol to produce (Z)-2-acetamido-N-phenyl-3-(phenyl/substituted phenyl)prop-2-enamides **2(a-l).**which were made to react with the schiff's bases (benzylidine/substituted benzylidine) anilines **3(a,b)** in the presence of triethylamine as a catalyst in ethanol medium under reflux condition for 2 h to produce 4-(benzylidene/substituted benzylidene)-N-arylamino-2-(styryl/substituted styryl)-1H-imidazole-5(4H)-one derivatives **6(a-l)** which were tested for their anti-bacterial activity. A reasonable mechanism has been formulated for the formation of these imidazoline-5-ones

**6(a-l) (scheme-2)**. The IR spectrum of the compound showed absence of absorptions for C=O and NH at 1740 $cm^{-1}$ and 3464 $cm^{-1}$ respectively and presence of absorptions for C=C, C-N and the $^{1}$H-NMR (DMSO-$d_6$)showed the absence of signals at at δ 5.9 and δ 8.2 for -NH protons and presence of signals for aromatic protons, two distinct signals for olefinic protons and signal for amide proton. $^{13}$C-NMR (DMSO-$d_6$) showed signals for -C=C, (-C=C) Ar, (C-N), (-C=N) and (C=O).The mass spectrum of the compounds **6(a-l)** showed the molecular ion peaks corresponding to the molecular weight of the compounds. Thus the structure of the novel compounds **6(a-l)** were confirmed by their spectral data.

**Scheme-1**




$Ar^2 = C_6H_5$

| 32 | a | b | c | d | e | f |
|---|---|---|---|---|---|---|
| Ar |  |  |  |  |  |  |
| $Ar^1$ | $C_6H_5$ | $C_6H_5$ | $C_6H_5$ | $C_6H_5$ | $C_6H_5$ | $C_6H_5$ |
| 32 | g | h | i | j | k | l |
| Ar |  |  |  |  |  |  |
| $Ar^1$ | 4-Cl-$C_6H_5$ | 4-Cl-$C_6H_5$ | 4-Cl-$C_6H_5$ | 4-Cl-$C_6H_5$ | 4-Cl-$C_6H_5$ | 4-Cl-$C_6H_5$ |

**Mechanism:**

The mechanism of the formation of one pot synthesis of 4-(benzylidene/substituted benzylidene)-N-aryl amino-2-(styryl/substituted styryl)-1H-imidazole-5(4H)-one derivatives and their anti- bacterial activity evaluation **6(a-l)** are given as follows **(scheme 1).** Initially nucleophilic addition of phenylhyrazine to 4-benzylidene-2-methyl oxazolin-5-ones **1(a-l)** which on treatment with schiff's bases and triethylamine produced (Z)-2-acetamido-N-phenyl-3-(phenyl/substituted phenyl)prop-2-enamides [**2(a-l)].2(a-l)** undergoes Michael type addition of the active methyl group of **2(a-l)** across the carbon-nitrogen double bond of the schiff's bases leading to the generation of styryl group by the loss of aniline. The resulting unstable intermediates readily undergo cyclocondensation to form stable imidazolin-5-one derivatives **6(a-l)**. The IR spectra of **6(a-l)** showed the presence of NH-stretching absorptions for NH and absence of stretching absorptions of NH-CO group. The $^{1}$H-NMR data showed only one signal for NH, which is $D_2O$ exchangeable and mass spectra of the compounds **6(a-l)** confirmed the molecular weight of the compounds.

These novel compounds 4-(benzylidene/substitutedbenzylidene)-N-arylamino-2-(styryl/substituted styryl)-1H-imidazole-5(4H)-one derivatives **6(a-l)** were tested for their anti-bacterial activity and produced impressive results.

**Scheme-2**

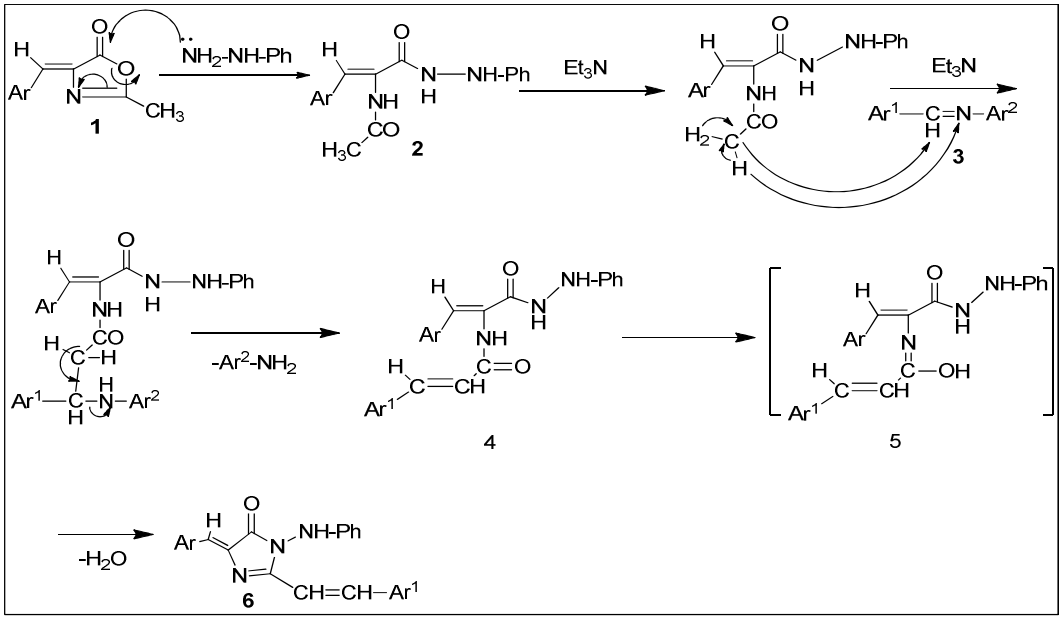



**Table-1**

Synthesis of [**2(a-l)]** from [**1(a-l)].**

| Entry | Starting material | Product | Time (min) | Yield* | M.P($^0$C) [lit. M.P $^o$C] | M. Wt |
|---|---|---|---|---|---|---|
| 1 | **1a** | **2a** | 60 | 80 | 158-160 | 295 |
| 2 | **1b** | **2b** | 60 | 80 | 164-166 | 325 |
| 3 | **1c** | **2c** | 65 | 78 | 153-155 | 313 |
| 4 | **1d** | **2d** | 60 | 80 | 156-158 | 340 |
| 5 | **1e** | **2e** | 70 | 75 | 164-166 | 329 |
| 6 | **1f** | **2f** | 60 | 80 | 156-158 | 329 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 7 | **1g** | **2g** | 65 | 81 | 158-160 | 357 |
| 8 | **1h** | **2h** | 70 | 80 | 163-165 | 388 |
| 9 | **1i** | **2i** | 65 | 82 | 153-156 | 375 |
| 10 | **1j** | **2j** | 70 | 82 | 157-159 | 403 |
| 11 | **1k** | **2k** | 60 | 81 | 163-167 | 374 |
| 12 | **1l** | **2l** | 70 | 80 | 156-158 | 374 |

***** Refers to yields of crude products only.

**Table-2**

Synthesis of [**6(a-l)]** from [**2(a-l)]** and [**3(a,b)]** after mp structure **6(a-l)**

| Entry | Starting material | Schiffs reagent | product **6(a-l)** molecular formula | Yield* | M.P($^0$C) |
|---|---|---|---|---|---|
| 1 | **2a** | **3(a,b)** | $C_{24}H_{19}N_3O$ | 65 | 164-166 |
| 2 | **2b** | **3(a,b)** | $C_{25}H_{21}N_3O_2$ | 63 | 165-167 |
| 3 | **2c** | **3(a,b)** | $C_{24}H_{18}N_3OF$ | 60 | 160-163 |
| 4 | **2d** | **3(a,b)** | $C_{24}H_{18}N_4O_3$ | 50 | 169-173 |
| 5 | **2e** | **3(a,b)** | $C_{24}H_{18}N_3OCl$ | 50 | 155-157 |
| 6 | **2f** | **3(a,b)** | $C_{24}H_{18}N_3OCl$ | 43 | 180-185 |
| 7 | **2g** | **3(a,b)** | $C_{24}H_{18}N_3OCl$ | 54 | 155-157 |
| 8 | **2h** | **3(a,b)** | $C_{25}H_{21}N_3O_2Cl$ | 65 | 172-174 |
| 9 | **2i** | **3(a,b)** | $C_{24}H_{18}N_3OFCl$ | 66 | 212-214 |
| 10 | **2j** | **3(a,b)** | $C_{24}H_{18}N_4O_3C1$ | 64 | 210-212 |

| | | | | | |
|---|---|---|---|---|---|
| 11 | **2k** | **3(a,b)** | $C_{24}H_{17}N_3OC1_2$ | 50 | 204-206 |
| 12 | **2l** | **3f(a,b)** | $C_{24}H_{17}N_3OC1_2$ | 48 | 211-213 |

***** Refers to yields of crude products only.

**Spectral analysis of(4Z)-4-benzylidene-1-methyl-2-styryl-1H-imidazol-5(4H)-one derivatives [6(a-l)]:**

**6a:** IR (KBr) $cm^{-1}$: 3444 (-NH), 1668 (-C=O), 3252(Ar),1595(C=C),1256(C-N). $^1$H- NMR (DMSO-$d_6$): δ 6.8 (d, 2H, HC=CH),δ7.4-8.0(m,15H, Ar-H), δ 8.2 (s, 1H, =CH),δ 8.4 (s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 116(-C=C), δ 119 (-C=C), δ 123(-C=C)Ar, δ 137(C-N), δ 141(-C=N), δ 167 (C=O). Mass (m/z)= 366 (M+1 )(100%).

**6b:** IR (KBr) $cm^{-1}$ :3436 (-NH), 1674 (-C=O), 3250(Ar),1590(C=C),1254(C-N). $^1$H- NMR (DMSO-$d_6$): δ 4.2 (s,3H,-$OCH_3$),δ 6.7 (d, 2H, HC=CH),δ 7.4-8.0 (m, 14H, Ar-H), δ 8.2 (s, 1H, =CH),δ 8.4 (s, 1H, NH).$^{13}$C- NMR (DMSO-$d_6$): δ 116 (-C=C), δ 118 (-C=C), δ 122 (-C=C)Ar, δ 138(C-N), δ 143 (-C=N), δ 166 (C=O). Mass (m/z) : 396 (M+1 )(100%).

**6c:** IR (KBr) $cm^{-1}$ : 3423 (-NH), 1661 (-C=O) 3166(Ar),1555(C=C),1262(C-N) $^1$H- NMR (DMSO-$d_6$): δ 6.9 (d, 2H, HC=CH),δ 7.4-8.0(m,14H,Ar-H), δ 8.1 (s, 1H, =CH), δ 8.4 (s, 1H, NH).$^{13}$C- NMR (DMSO-$d_6$): δ 117 (-C=C), δ 118 (-C=C), δ 121 (-C=C)Ar, δ 137 (C-N), δ 144 (-C=N), δ 166 (C=O).Mass (m/z) : 385 (M+1 )(100%).

**6d:** IR (KBr) $cm^{-1}$ : 3455 (-NH), 1662 (-C=O) 3064(Ar),1598(C=C),1272(C-N) $^1$H- NMR (DMSO-$d_6$): δ 6.8 (d,2H,HC=CH),δ 7.5-8.0(m,14H,Ar-H), δ 8.0 (s, 1H, =CH), δ 8.4 (s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 116 (-C=C), δ 118 (-C=C), δ 123 (-C=C)Ar, δ 138 (C-N), δ 145 (-C=N), δ 167 (C=O). Mass (m/z) : 411 (M+1 )(100%).

**6e:** IR (KBr) $cm^{-1}$ : 3434 (-NH), 1693 (-C=O) 3146(Ar),1570(C=C),1265(C-N). $^1$H- NMR (DMSO-$d_6$):δ 6.9 (d, 2H, HC=CH),δ 7.5-8.0(m,14H, Ar-H), δ 8.2 (s, 1H, =CH), δ 8.4 (s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 117 (-C=C), δ (-C=C), δ 124 (-C=C)Ar, δ 137 (C-N), δ 144 (-C=N), δ 168 (C=O). Mass (m/z) : 400 (M+1 )(100%).

**6f:** IR (KBr) $cm^{-1}$ : 3434 (-NH), 1653 (-C=O) 3250(Ar),1593(C=C),1254(C-N) $^1$H- NMR (DMSO-$d_6$): δ 6.8 (d, 2H, HC=CH),δ7.4-7.9(m,14H, Ar-H), δ 8.1 (s, 1H, =CH), δ 8.4 (s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 116 (-C=C), δ 118 (-C=C), δ 123 (-C=C)Ar, δ 138 (C-N), δ 145 (-C=N), δ 167 (C=O). Mass (m/z) : 400 (M+1 )(100%)

**6g:** IR (KBr) $cm^{-1}$ : 3401 (-NH), 1699 (-C=O) 3220(Ar),1541(C=C),1286(C-N) $^1$H-NMR (DMSO-$d_6$): δ 6.9 (d, 2H, HC=CH),δ 7.4-7.9(m,14H, Ar-H), δ 8.2 (s, 1H, =CH), δ 8.4

(s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 117 (-C=C), δ 119 (-C=C), δ 122 (-C=C)Ar, δ 137 (C-N), δ 144 (-C=N), δ 166 (C=O). Mass (m/z) : 400 (M+1 )(100%).

**6h:** IR (KBr) cm$^{-1}$ : 3425 (-NH), 1664 (-C=O) 3247(Ar),1595(C=C),1254(C-N). $^1$H-NMR (DMSO-$d_6$): δ 4.3 (s, 3H, -$CH_3$),δ 6.8 (d, 2H, HC=CH), δ 7.4-7.9 (m, 13H, Ar-H), δ 8.2 (s, 1H, =CH), δ 8.3 (s, 1H, NH). $^{13}$C- NMR ( DMSO-$d_6$): δ 117 (-C=C), δ 120 (-C=C), δ 122 (-C=C)Ar,δ 138 (C-N), δ 146 (-C=N), δ 168 (C=O). Mass (m/z) : 430 (M+1 )(100%).

**6i:** IR (KBr) cm$^{-1}$ : 3434 (-NH), 1664 (-C=O) 3251(Ar),1594(C=C),1252(C-N) .$^1$H-NMR (DMSO-$d_6$): δ 6.9 (d, 2H, HC=CH),δ 7.4-8.1(m,13H,Ar-H), δ 8.1 (s, 1H, =CH),δ 8.4 (s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 118 (-C=C), δ 119 (-C=C), δ 121 (-C=C)Ar, δ 137 (C-N), δ 146 (-C=N), δ 168 (C=O). Mass (m/z) : 418 (M+1 )(100%).

**6j:** IR (KBr) cm$^{-1}$ : 3428 (-NH), 1650 (-C=O) 3250(Ar),1593(C=C),1255(C-N). $^1$H-NMR (DMSO-$d_6$):δ 6.6 (d, 2H, HC=CH),δ7.4-8.1(m,13H, Ar-H), δ 8.2 (s, 1H, =CH),δ 8.4 (s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 116 (-C=C), δ 118 (-C=C), δ 123 (-C=C) Ar, δ 139 (C-N), δ 147 (-C=N), δ 167 (C=O). Mass (m/z) : 445 (M+1 )(100%).

**6k:** IR (KBr) cm$^{-1}$ : 3440 (-NH), 1662 (-C=O) 3247(Ar),1592(C=C),1254(C-N). $^1$H-NMR (DMSO-$d_6$): δ 6.8 (d, 2H, HC=CH), δ7.4-8.0(m, 13H, Ar-H), δ 8.1 (s, 1H, =CH),δ 8.4 (s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 117 (-C=C), δ 119 (-C=C), δ 124 (-C=C) Ar, δ 138 (C-N), δ 148 (-C=N), δ 168 (C=O). Mass (m/z): 434 (M+1 )(100%).

**6l:** IR (KBr) cm$^{-1}$ : 3430 (-NH), 1665 (-C=O) 3251(Ar),1594(C=C),1252(C-N). $^1$H-NMR (DMSO-$d_6$): δ 6.7 (d, 2H, HC=CH),δ7.4-8.0(m,13H, Ar-H), δ 8.1 (s, 1H, =CH),δ 8.4 (s, 1H, NH). $^{13}$C- NMR (DMSO-$d_6$): δ 117 (-C=C), δ 119 (-C=C), δ 123 (-C=C) Ar, δ 138 (C-N), δ 147 (-C=N), δ 166 (C=O). Mass (m/z) : 434 (M+1) (100%).

**In Vitro Anti-bacterial activity:**

The synthesized compounds [**6(a-l)]** were screened for their in vitro antibacterial activity aganist *Escherichia coli* (NCIM 2065*), Providencia aeruginosa* (NCIM 2200), *Pseudomonas azotogensis* (NCIM 2075) and *Baccilus Subtillis* (NCIM 2063). Antibacterial activity of compounds was evaluated using agar-well diffusion method[17]. The petriplates were sterilized using an autoclave at 120 $^o$C for 30 min. A petri-dish of 100 mm diameter was filled with 50 ml of freshly prepared Nutrient Agar media and allowed to solidify. Different bacterial species were inoculated on to the medium by streak plate method. The plates were incubated at 30$^o$C temperature and zone of inhibition was measured after 24 h. The standard used for determining the antibacterial activity is streptomycin. The compounds were

dissolved in DMF and activity described at 100 µg/ml level. From the data presented in **Table 3**, it is clear that compounds **6c, 6e, 6f, 6i, 6k and 6l** possess good activity against *Escherichia coli* and *Baccilus Subtillis*. Other compounds exhibited moderate antibacterial activity against *E.coli*. However, all compounds showed moderate activity against, *Providencia aeruginosa* and *Pseudomonas azotogensis.* **(Table-3).**

**Table-3**

Anti-bacterial activity of {**6(a-l)}.**

| S.No. | Compound | *Escherichia coli* | *Providencia aeruginosa* | *Pseudomonas azotogensis* | *Baccilus subtillis* |
|---|---|---|---|---|---|
| 1 | **6a** | 10 | 11 | 06 | 12 |
| 2 | **6b** | 7 | 02 | 07 | 14 |
| 3 | **6c** | 23 | 13 | 11 | 19 |
| 4 | **6d** | 10 | 14 | 08 | 13 |
| 5 | **6e** | 21 | 06 | 11 | 22 |
| 6 | **6f** | 22 | 02 | 10 | 23 |
| 7 | **6g** | 06 | 11 | 06 | 15 |
| 8 | **6h** | 10 | 06 | 11 | 12 |
| 9 | **6i** | 22 | 10 | 15 | 15 |
| 10 | **6j** | 03 | 02 | 03 | 12 |
| 11 | **6k** | 15 | 08 | 03 | 18 |
| 12 | **6l** | 21 | 12 | 04 | 18 |
| 13 | **Streptomycin** | **30** | **32** | **28** | **30** |

**Conclusion:**

All the new imidazole derivatives **6(a-l)** were synthesized with high purities, good yields and minimum reaction times by making use of ecofriendly chemicals and simple processing mechanisms.

The compounds **6(c), 6(e), 6(f), 6(i), 6(k), 6(l)** exhibited good activity against *Escherichia coli* and *Baccilus Subtillis***.** Remaing all compounds showed no antibacterial activity against *Escherichia coli* and *Baccilus Subtillis***.** The imidazol-5-one derivatives **6(a-l)** showed no activityagainst, *Providencia aeruginosa* and *Pseudomonas azotogensis.*

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