ISBN 978-90005-44-4

A One Day National Level Conference
On

"Multi-disciplinary Research Methodology In Humanities, Languages & Literature, Commerce & Management Science, Science & Technology

17th April, 2023

Editors

Dr.P.P. Sharma, Dr.R.P. Pawar,

Dr.S.U. Tekale

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P.B. No. 131, Gokulwadi, Aurangapura, Aurangabad.
Tel. No. 0240-2329204,Mob:- 09970067971.
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Type Setter:-
Kirti Digitals, Aurangabad.
Mobile:- 09403536655.

Edition:- 2023

INDEX

ULTRASOUND PROMOTED ONE-POT SYNTHESIS OF SUBSTITUTED PYRAZOLES

Chandrashekhar G. Devkate[*1], Ajay M. Patil[2], Satish Kola[3], Mohammad Idrees M. Siddique[4]

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ABSTRACT

Synthesis ofsubstituted pyrazoles by one pot condensation reaction of substituted cinnamaldehydes and tosylhydrazine in the presence of glyoxy;ic acid under solvent free condition. The methodology highlights the use of ultrasonic irradiation as non conventional sources. The catalyst used is readily available and cost effective which makes the method more green and efficient.

Key words: 3-Substituted pyrazole,Glyoxylic acid, Ultrasound irradiation.

INTRODUCTION

Pyrazoles and its derivatives are usually used in medicinal chemistry as they havelarge range of biological and pharmacological activities such asanti-inflammatory, analgesic, antibacterial,
antidiabetic, antipyretic, antiviral,uricosuric, hypoglycemic, antineoplastic antiarthritic, and antiphlogistic properties[1–4].Due to various important features of pyrazolesvarious synthetic methods are reported for the pyrazole synthesis.Condensation of hydrazonyl halides with b-dicarbonyl compounds and 1,3-dipolar cycloaddition of diazo compounds with alkynes[5–7] are found to yield pyrazoles. The most usually used synthetic protocol for obtainingpolysubstitutedpyrazoles is by condensation of 1,3-dicarbonyl compounds with hydrazines using acid catalysts like sulphuric acid[8], polystyrensulphonic acid[9], ionic liquid[10] and hydrochloric acid[11].
Here we are interested to use glyoxalic acid as it is a strong acid with excessive large applications such asDiels Alder reaction[12], deportation of oximes[13] and for the synthesis of imidazoles[14].

MATERIALS AND METHODS

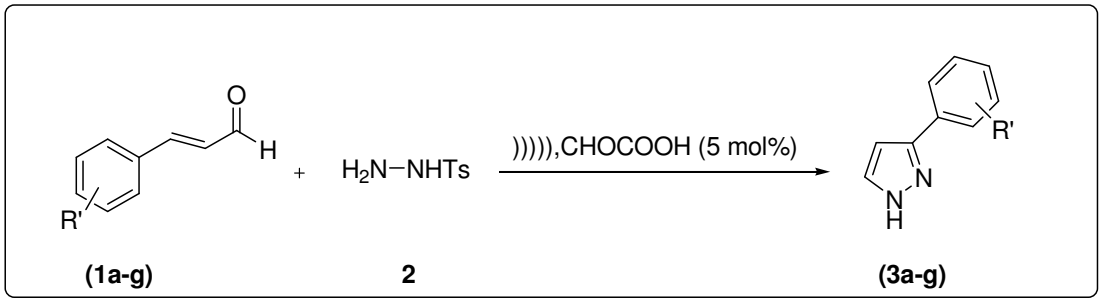
General procedure for the synthesis of Pyrazoles

Cinnamaldehyde (**1**) (1.00mmol) and tosylhydrazine (**2**) (1.00 mmol) was taken in RBF to that glyoxalic acid (5 mol%) was addedand then after the RBF was kept into the ultrasonic water bath, and was irradiated at 40% of the power of the ultrasonic bath at rt. By using TLC the progress of the reaction was monitor. After complete conversion the reaction mass was poured on crushed ice. The obtained solids were filtered, washed with water and dried. The crude compounds were crystallized using (1:1) DMF-Ethanol.

Spectral data for representative compound 3a.

white solid, FTIR cm^{-1}: 3165 (N-H str.), 1536 (C=N str., Pyrazolyl), 1048 (C-O str.);^{1}H-NMR (400 MHz, DMSO): δ 3.77 (s, 3H, -OCH_3), 6.65 (d, 1H, Ar-H, J = 8 Hz), 7.28 (t, 1H, Ar-H, J = 8 Hz), 7.37 (d, 1H, Ar-H, J = 8 Hz), 7.44 (s, 1H, Ar-H), 7.66 (s, 2H, Pyrazolyl), and 14.02 (s, 1H, N-H) ppm;^{13}C-NMR (100 MHz, DMSO): δ 159.58, 133.87,

129.59, 117.65, 112.90, 110.50, 101.98, 54.81 ppm; MS (ESI, m/z): calcd for $C_{10}H_{10}N_{2O}$ (M + H^+) 174.0793; found: 175.1162.

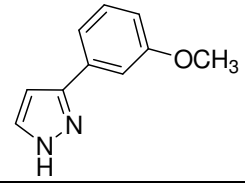
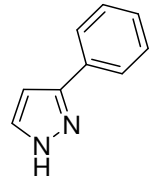
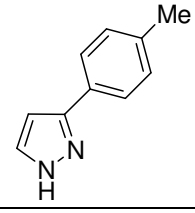


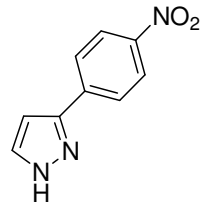
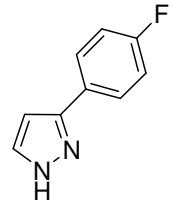
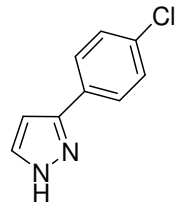
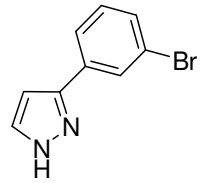
Scheme: Synthesis of substituted pyrazoles (**3a-g**) usingglyoxalic acidunder ultrasound irradiated.

RESULTS AND DISCUSSION

The synthesis of pyrazoleusing readily available starting materials such as cinnamaldehyde (**1a-g**) andp-toluenesulfonyl hydrazide ($TsNHNH_2$) (**2**). The use of glyoxalic acid as a catalyst and media for the synthesis makes the method more cost effective. Here, we have noted that the conversion takes place in less time with respect to cinnamaldehyde as the donating group increasing and as we have noticed that if there is any strong withdrawing group present than the conversion is lees (**Table 1, 3d**).The reactions were carried out at room temperature for 30 min.The progress of the reaction was monitored by TLC. Various cinnamaldehydes (**1a-g)** could give target pyrazoles through the same action (**3a-g**).And the use of ultrasound irradiation as a non-conventional source has played a key role in the synthesis as compared to other conventional methods.

Table 1: Glyoxalic acid catalyzed synthesis of pyrazoles[a].

Entry	R'	Product	Yield	M. P. (℃)
3a	m-OMe		93	91-92
3b	-H		86	77-81
3c	p-Me		90	75-77

3d	p-NO_2		65	195-196
3e	p-F		75	102-104
3f	p-Cl		88	100-104
3g	m-Br		85	74-76

CONCLUSION

In conclusion, we have investigated a simple, highly efficient, and environmentally friendly method for the synthesis of substituted pyrazoles. Here, the use of glyoxalic acid works as anexcellent catalyst. The use of ultrasound irradiation as a non-conventional source has played a key role in the synthesis. And the further use of the methodology for the synthesis of other useful heterocycles is going on our laboratory.

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"AN EFFICIENT SYNTHESIS OF SOME NOVEL BIO ACTIVE 5-OXO-IMIDAZOLINE DERIVATIVES COMPRISING QUINOLINEBENZOFURAN AND PYRAZOLE MOIETY"

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ABSTRACT

In the present work we have reported synthesis of novel 5-oxo-imidazoline **(5a-e)** derivatives containing benzofuran, pyrazole, quinoline ether moieties. It comprises preparation of intermediate 4-((2-(*p*-tolyloxy)-substituted quinolin-3-yl) methylene)-2-phenyloxazol-5(4*H*)-one **(3a-e)** from benzoyl glycine and substituted 2-(*p*-tolyloxy)-substituted quinoline-3-carbaldehyde **(2a-e)**in presence anhydrous sodium acetate and acetic anhydride. These oxazolinone derivatives **(3a-e)** is further treated with 5-(benzofuran-2-yl)-1-phenyl-1*H*-pyrazole-3-carbohydrazide **(4)** in acetic acid to afford the target derivatives 4-((2-(*p*-tolyloxy)-substitutedquinolin-3-yl)methylene)-4,5-dihydro-5-oxo-2-phenylimidazol-1-yl)-5-(benzofuran-2-yl)-1-phenyl-1*H*-pyrazole-3-carboxamide **(5a-e)**.The characterization of newly synthesized compound **(3a-e)** and **(5a-e)** was made by, FTIR, ^{1}HNMR, ^{13}CNMR, elemental analysis and further supported by Mass spectra. All synthesized compounds were screened for their *in-vitro* antimicrobial activity at different concentration against a panel of pathogenic microorganism including *S. aureus* as Gram positivewhile *E.coli, P.vulgaris, S.typhi* as Gram negative bacterial strains. The result of bioassay is compared with Chloramphenicol as standard drug.

Keywords: Carbohydrazide, 5-oxo-imidazoline, Phenyl oxazole, (*p*-tolyloxy)quinoline-3-carbaldehyde, Antimicrobial activity.

INTRODUCTION

The Nitrogen containing heterocyclic compounds such as imidazolinone have grown enormous importance due to biologically accepted pharmacophores owing to wide range of biological activities as well as their various pharmacological actions. Consequently over the year's5-oxo-imidazoline related drugs have fascinated the attention of the scientific community to synthesize a large number of imidazolinone derivatives as novel chemotherapeutic medicines. Numerous drugs contain imidazole ring, such as antifungal drugs like Ketoconazole, Miconazole, Clotrimoxazole and Nitroimidazole. The significance

of imidazolinone with quinoline heterocyclic nucleus in the field of medicinal chemistry research is worth mentioning. 5-oxo-imidazole have been reported to exhibit a wide range of diverse bioactivities such as antimicrobial[1-7], non-purine xanthine oxidase inhibitors[8],anticancer[9-11], antihistaminic[12],antioxidant agents[13], antifungal[14,15], antipyretic and wound healing[16], anticonvulsant[17,18], biological[19-22],anthhelmthetic[23],photochemical probe agents[24], anti-hyperglycaemic agents[25], CNS depressant[26],herbicidal[27], anti-HIV[28], multi-domain peptide[29]. Besides this Imidazole nucleus is also present in natural products such as,alkaloids[30], they are utilized as valuable synthetic templates for the preparation of innovative compounds with specific biological, pharmaceutical and material properties.

All of these above facts inspired us to synthesize some novel series of 5-(benzofuran-2-yl)-*N*-4,5-dihydro-5-oxo-4-((2-phenoxyquinolin-3-yl)methylene)-2-phenylimidazol-1-yl)-1-methyl-1*H*-pyrazole-3-carboxamide **5a-e** derivatives and screen them against some pathogenic bacterial strains with a assumed that combination of imidazolinone ring with quinoline moiety may enhance their pharmacological activities as in the area of medicinal chemistry the synthesis of these types of derivatives is always a crucial factor.

MATERIAL &METHODS

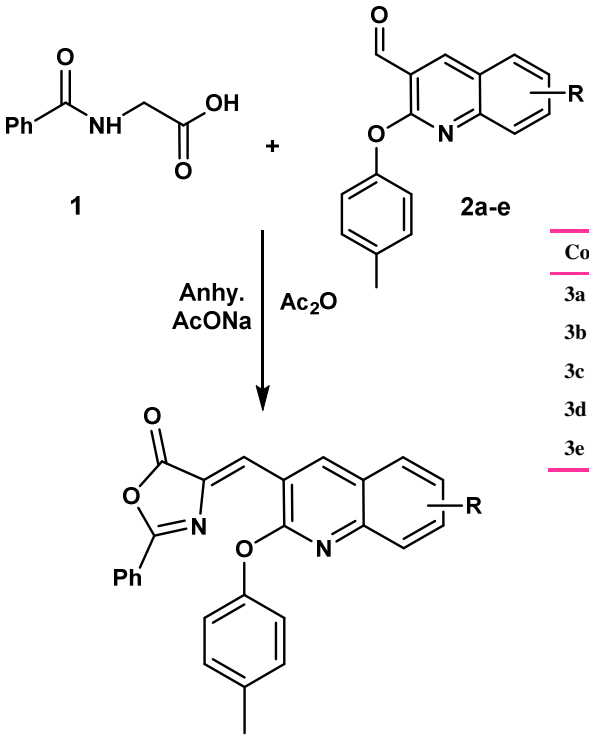
Chemicals used for the synthesis were of AR grade of Merck, S.D. Fine and Aldrich. The reactions were monitored by E. Merck TLC aluminum sheet silica $gel_{60}F_{254}$ and visualizing the spot in UV Cabinet and iodine chamber. The melting points were recorded in open capillary in paraffin bath and are uncorrected. ^{1}H NMR spectra are recorded on a Bruker AM 400 instrument (400 MHz) using tetramethylsilane (TMS) as an internal reference and DMSO-*d6*as solvent. Chemical Shifts are given in parts per million (ppm). Positive-ion Electro Spray Ionization (ESI) mass spectra were obtained with a Waters Micromass Q–TOF Micro, Mass Spectrophotometer. IR spectra were recorded on a Shimadzu IR Spectrophotometer (KBr, υ_{max} in cm^{-1}). The compounds are purified by using column chromatography on silica gel (60-120 mesh). Elemental (CHN) analysis was done using Thermo Scientific (Flash-2000), the compounds were analysed for carbon, hydrogen and nitrogen and the results obtained are in good agreement with the calculated values.

Procedure for the synthesis of 4-((2-(*p*-tolyloxy)-substituted quinolin-3-yl) methylene)-2-phenyloxazol-5(4*H*)-one(3a-e): In a 250mL conical flask mixture of (2.73g , 0.015mol) of benzoyl glycine[31](**1**) and (4.15g , 0.015mol) of 2-(*p*-tolyloxy)-8-methylquinoline-3-carbaldehyde[32] (**2a)** was taken to that (2.12mL, 0.045mol) acetic anhydride and (1.23g , 0.015mol) anhydrous sodium acetate were added. Reaction content was heated on electric hot plate with constant shaking until the mixture liquefies completely, then it was refluxed on water bath for 2h then 10mL of ethanol was added to the content of the flask and the mixture was allowed to stand overnight. The crystalline precipitate formed was filtered, washed, dried and recrystallized using benzene to afford compound **3a.**Correspondingly, other (4)-4-((2-(*p*-tolyloxy)-substituted quinolin-3-yl) methylene)-2-phenyloxazol-5(4*H*)-ones **3b-e**were synthesized from compound **1**and **2b-e** by following the same procedure for **3a 4-((2-(*P*-Tolyloxy)-8-methyl quinoline) methylene)-2-phenyloxazol-5(4*H*)-one(3a)**: yellow amorphous solid, mp 198°C; yield, 85%; (from benzene); M.F; $C_{27}H_{20}N_2O_3$.,IR(KBr, υ_{max} in cm^{-1}):3063,3035(C-H str., arom.), 2952, (C-H asym. str., aliph.), 2919(C-H sym. str., aliph.), 1469,1451 (C-H asym.def., aliph.), 1369(C-H sym.def., aliph.), 1602,1553 (C=C str., arom.), 1075 (C-H i.p.def., arom.), 884,759(C-H o.o.p.def., arom.), 1251(C-O-C asym. str., ether), 1053,1022 (C-O-C sym. str., ether), 1656,1602 (C=N str., oxazolone ring), 1221(C-N str.), 1765,1796(CO str. in oxazolone ring).^{1}H NMR (DMSO-d6) δppm;2.34(s, 3H, -C<u>H</u>$_3$attached to aromatic ring), 2.37(s,3H, -C<u>H</u>$_3$ attached to quinoline ring), 7.10-8.29(m,14H, aromatic & quinoline ring protons).

4-((2-(*P*-Tolyloxy)-7-methylquinolin-3-yl)methylene)-2-phenyloxazol-5(4*H*)-one(3b): yellow amorphous solid, mp 203°C; yield, 83%; (from benzene); M.F; $C_{27}H_{20}N_2O_3$. IR(KBr, υ_{max} in cm^{-1}): 3061, 3032(C-H str., arom.), 2954, (C-H asym. str., aliph.), 2916(C-H sym. str., aliph.), 1455, 1470(C-H asym.def., aliph.), 1366(C-H sym.def., aliph.), 1608, 1556(C=C str., arom.), 1072(C-H i.p.def., arom.), 886,756(C-H o.o.p.def., arom.), 1253(C-O-C asym. str., ether), 1051,1024 (C-O-C sym. str., ether), 1652, 1606(C=N str., oxazolone ring), 1226(C-N str.), 1763,1793(CO str. in oxazolone ring). ^{1}H NMR (DMSO-d6) δppm;2.35(s, 3H, -C<u>H</u>$_3$ attached to aromatic ring), 2.34(s, 3H, -C<u>H</u>$_3$ attached to quinoline ring), 7.10-8.50(m, 14H, aromatic & quinoline ring protons).

4-((2-(*P*-Tolyloxy)-6-methylquinolin-3-yl)methylene)-2-phenyloxazol-5(4*H*)-one (3c): yellow amorphous solid, mp 196°C; yield, 88%; (from benzene); M.F $C_{27}H_{20}N_2O_3$. IR(KBr, υ_{max} in cm^{-1}):3064, 3037(C-H str., arom.), 2956(C-H asym. str., aliph.), 2921(C-H sym. str., aliph.), 1453, 1473(C-H asym.def., aliph.), 1363(C-H sym.def., aliph.), 1609,1558 (C=C str., arom.), 1071(C-H i.p.def., arom.), 881,761(C-H o.o.p.def., arom.), 1253(C-O-C asym. str., ether), 1056,1027 (C-O-C sym. str., ether), 1659,1609(C=N str., oxazolone ring), 1230(C-N str.), 1767,1791(CO str. in oxazolone ring). ^{1}H NMR (DMSO-d6) δppm;2.34(s, 3H, -C<u>H</u>$_3$ attached to aromatic ring), 2.35(s,3H, -C<u>H</u>$_3$ attached to quinoline ring), 6.90-8.44(m,14H, aromatic & quinoline ring protons).

Reaction scheme – I



Code	-R
3a	8-CH_3
3b	7-CH_3
3c	6-CH_3
3d	6-Br
3e	6-OCH_3

(3a-e)

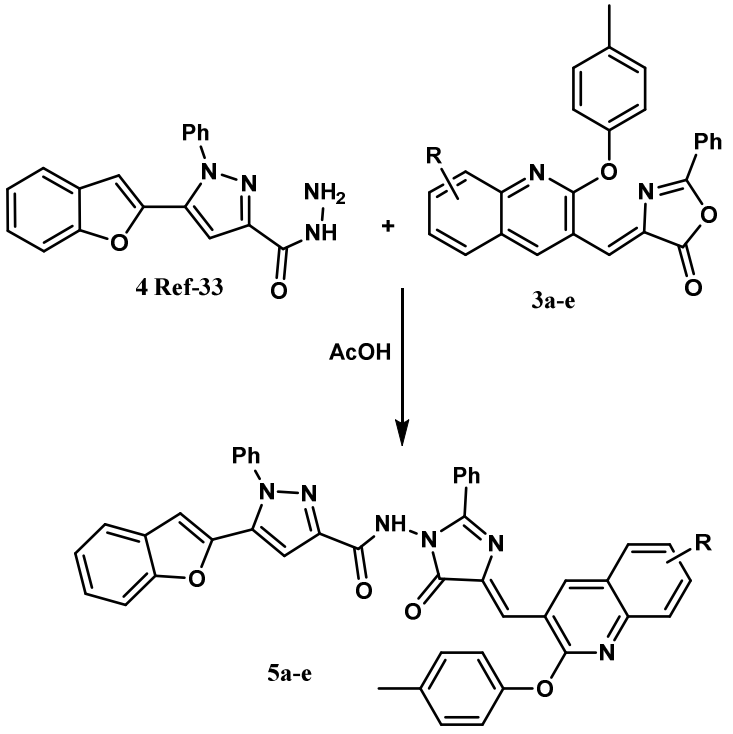
4-((2-(*P*-Tolyloxy)-6-bromoquinolin-3-yl)methylene)-2-phenyloxazol-5(4*H*)-one (3d): yellow amorphous solid, mp 201°C; yield, 86 %; (from benzene); M.F $C_{26}H_{17}BrN_2O_3$.IR(KBr, υ_{max} in cm^{-1}): 3066, 3038(C-H str., arom.), 2951 (C-H asym. str., aliph.), 2922(C-H sym. str., aliph.), 1453, 1476(C-H asym.def., aliph.), 1366(C-H sym.def., aliph.), 1605,1550(C=C str., arom.), 1078(C-H i.p.def., arom.), 884, 754(C-H o.o.p.def., arom.), 1254(C-O-C asym. str., ether), 1050,1020(C-O-C sym. str., ether), 1658,1605 (C=N

str., oxazolone ring), 1221(C-N str.), 1765,1796(CO str. in oxazolone ring). ^{1}H NMR (DMSO-d6) δppm;2.36(s, 3H, -C<u>H$_3$</u> attached to aromatic ring), 6.89-8.48(m, 14H, aromatic & quinoline ring protons).

4-((2-(p-tolyloxy)-6-methoxyquinolin-3-yl)methylene)-2-phenyloxazol-5(4H)-one(3e):: Yellow amorphous solid, mp 204°C; yield, 84 %; (from benzene); M.F C27H20N2O4. IR(KBr, υmax in cm-1): 3060,3033(C-H str., arom.), 2956(C-H asym. str., aliph.), 2924(C-H sym. str., aliph.), 1456,1462(C-H asym.def., aliph.), 1365(C-H sym.def., aliph.), 1604, 1556(C=C str., arom.), 1077 (C-H i.p.def., arom.), 882,763(C-H o.o.p.def., arom.), 1257(C-O-C asym. str., ether), 1053,1029 (C-O-C sym. str., ether), 1651,1604(C=N str., oxazolone ring), 1229(C-N str.), 1766, 1794(CO str. in oxazolone ring).

Procedure for the Synthesis of 5-(Benzofuran-2-yl)-*N*-(5-oxo-2-phenyl-4-((2-(*p*-tolyloxy)-substituted-quinolin-3-yl)methylene)-4,5-dihydroimidazol-1-yl)-1-phenyl-1*H*-pyrazole-3-carboxamide (5a-e): In100 mLR. B flask mixture of(2.18g, 0.005mol) of 4-((2-(P-Tolyloxy)-8-methyl quinoline) methylene)-2-phenyloxazol-5(4H)-one**(3a)** and (1.59 g, 0.005mol) of 5-(benzofuran-2-yl)-1-phenyl-1*H*-pyrazole-3-carbohydrazide **(4)**[33] was taken and 20mL of acetic acid was added, contents was refluxed for 8h. Resulting mass was poured into crushed ice, filtered and the product wasrecrystallized from ethanol to afford **(5a).**

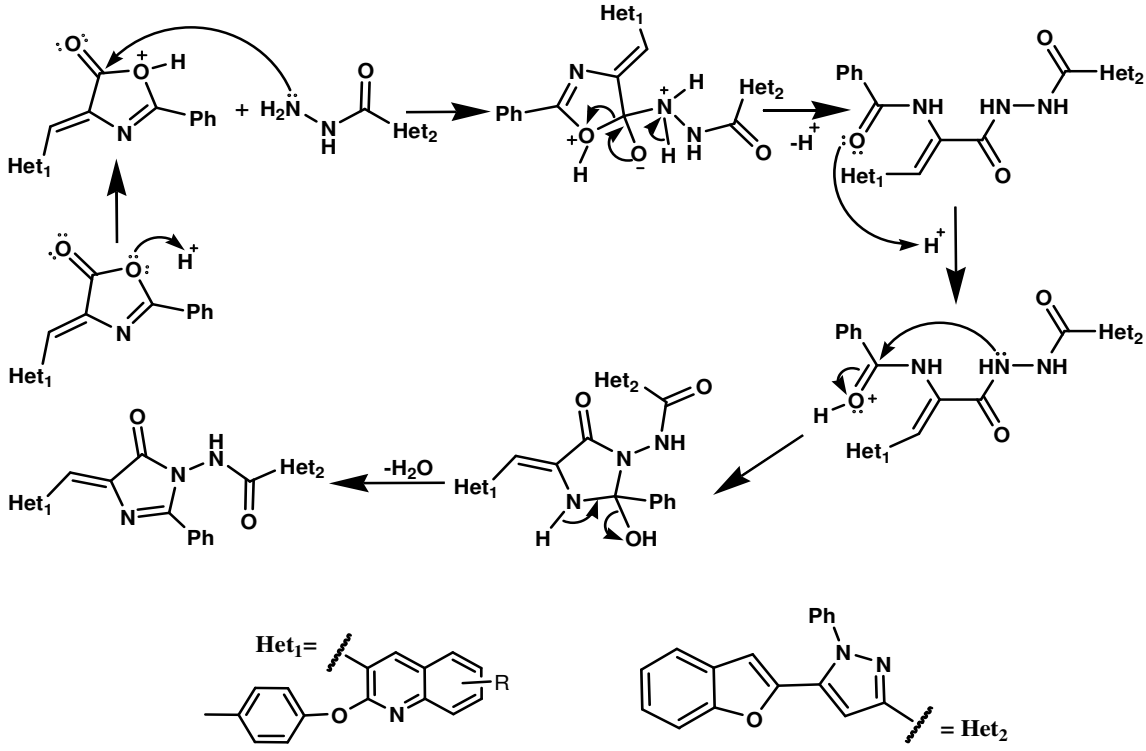
Reaction scheme -II



Code	-R
5a	8-CH_3
5b	7-CH_3
5c	6-CH_3
5d	6-Br
5e	6-OCH_3

Correspondingly, other 4-((2-(*p*-tolyloxy)-substitutedquinolin-3-yl)methylene)-4,5-dihydro-5-oxo-2-phenylimidazol-1-yl)-5-(benzofuran-2-yl)-1-phenyl-1*H*-pyrazole-3-carboxamide**5b-e**were also synthesized from compound **4**and **3a-i** by following the similar procedure for **5a**.

Plausible Mechanism:



4-((2-(*P*-Tolyloxy)-8-methylquinolin-3-yl)methylene)-4,5-dihydro-5-oxo-2-phenylimidazol-1-yl)-5-(benzo furan-2-yl)-1-phenyl-1*H*-pyrazole-3-carboxamide (5a): IR(KBr, υ_{max} in cm^{-1}): 3408, 3198(N-H str., -CONH-), 3063(C-H str., arom.), 2955(C-H asym. str., aliph.), 2841(C-H sym. str., aliph.), 1454(C-H asym.def., aliph.), 1370(C-H sym.def., aliph.), 1506, 1453(C=C str., arom.), 1075, 1023,1006(C-H i.p.def., arom.), 836(C-H o.o.p.def., arom.), 1258, 1235(C-O-C asym. str., ether), 1075, 1023(C-O-C sym. str., ether), 1525(C=N str., pyrazole ,imidazole and quinoline nucleus), 1659(C=O str., 5-oxo-imidazolines ring), 1619(C=O str., amide group), 1166(C-N-C str.), 1075(C-N str.). ^{1}H NMR (DMSO-d_6, 400 MHz):δ (ppm)2.40(s, 3H, -C<u>H</u>$_3$ attached to aromatic ring), 2.38(s, 3H, -C<u>H</u>$_3$ attached to quinoline ring), 12.22(s,1H, -CON<u>H</u>- linkage), 6.57-8.90(m,25H, aryl ,pyrazole, quinoline ring and ethylenic protons),^{13}C NMR (DMSO-d_6): δ (ppm)23(-CH_3), 20(-CH_3 attached to quinoline ring), 106.51, 110, 119, 120, 123, 125, 126, 127, 129 ,130, 132, 133, 135, 139, 140, 144(s,1C, C_3 of pyrazole ring), 154(s,1C, C_9 of Benzofuran ring), 155 (s,1C,C6 of quinoline),161(s, 1C,amide linkage), 166(s, 1C, C_5 of 5-oxo-imidazoline ring), 172(s, 1C, C_2 of quinoline ring to which phenyloxy group attached)GC-MS (*m/z)*:720 [M],Elemental Anal.Calcd for$C_{45}H_{32}N_6O_4$ calculated;C, 74.99; H, 4.47; N, 11.66; Found C, 74.79; H,4.33; N,11.34.

5-(Benzofuran-2-yl)-*N*-(4-((7-methyl-2-(*p*-tolyloxy)quinolin-3-yl)methylene)-5-oxo-2-phenyl-4,5-dihydroimida- zol-1-yl)-1-phenyl-1*H*-pyrazole-3-carboxamide(5b):IR(KBr, υ_{max} in cm^{-1}): 3405, 3193(N-H str., -CONH-), 3058(C-H str., arom.), 2953(C-H asym. str.,aliph.), 2844(C-H sym. str., aliph.), 1452(C-H asym.def., aliph.), 1367(C-H sym.def., aliph.), 1506, 1451(C=C str., arom.), 1074, 1032,1006(C-H i.p.def., arom.), 832(C-H o.o.p.def., arom.), 1255, 1232(C-O-C asym. str., ether), 1074,1032(C-O-C sym. str., ether), 1526(C=N str., Pyrazole ,imidazole and quinoline nucleus), 1656(C=O str., 5-oxo-imidazolines ring), 1617(C=O str., amide group) , 1161(C-N-C str.), 1074(C-N str.). ^{1}H NMR

(DMSO-d_6, 400 MHz):δ (ppm)2.43(s, 3H, -C<u>H</u>$_3$ attached to aromatic ring), 2.35(s, 3H, -C<u>H</u>$_3$ attached to quinoline ring), 12.16(s,1H, -CON<u>H</u>- linkage), 6.8-8.90(m,25H, aryl ,pyrazole, quinoline ring and ethylenic protons),^{13}C NMR (DMSO-d_6): δ (ppm)24(-CH_3), 20(-CH_3 attached to quinoline ring), 106, 114, 118, 121, 122, 125, 126, 127, 129 ,130, 132, 134, 135, 139, 140, 140(s,1C, C_3 of pyrazole ring), 155(s,1C, C_9 of Benzofuran ring), 154 (s,1C,C_6 of quinoline),160(s, 1C,amide linkage), 167(s, 1C, C_5 of 5-oxo-imidazoline ring), 170(s, 1C, C_2 of quinoline ring to which phenyloxy group attached)GC-MS (*m/z)*:720 [M],Elemental Anal. Calcd for$C_{45}H_{32}N_6O_4$calculated;C, 74.99; H, 4.47; N, 11.66; found C, 74.70; H, 4.38; N, 11.24.

5-(Benzofuran-2-yl)-*N*-(4-((6-methyl-2-(*p*-tolyloxy)quinolin-3-yl)methylene)-5-oxo-2-phenyl-4,5-dihydro imidazol-1-yl)-1-phenyl-1*H*-pyrazole-3-carboxamide (5c):IR(KBr, υ_{max} in cm^{-1}): 3407, 3199(N-H str., -CONH-), 3064(C-H str., arom.), 2956(C-H asym. str., aliph.), 2846(C-H sym. str., aliph.), 1455(C-H asym.def., aliph.), 1364(C-H sym.def., aliph.), 1500, 1457(C=C str., arom.), 1073, 1023, 1006(C-H i.p.def., arom.), 835(C-H o.o.p.def., arom.), 1251, 1234(C-O-C asym. str., ether), 1073, 1023(C-O-C sym. str., ether), 1528(C=N str., pyrazole, imidazole and quinoline nucleus), 1658(C=O str., 5-oxo-imidazolines ring), 1615(C=O str., amide group), 1168(C-N-C str.) , 1073(C-N str.). ^{1}H NMR (DMSO-d_6, 400 MHz):δ (ppm)2.40(s, 3H, -C<u>H</u>$_3$ attached to aromatic ring), 2.37(s, 3H, -C<u>H</u>$_3$ attached to quinoline ring), 12.20(s,1H, -CON<u>H</u>- linkage), 6.57-8.90(m,25H, aryl ,pyrazole, quinoline ring and ethylenic protons),^{13}C NMR (DMSO-d_6): δ (ppm)23(-C<u>H</u>$_3$), 21(-CH_3 attached to quinoline ring), 105, 111, 119, 121, 123, 124, 126, 127, 129 ,130, 132, 133, 135, 139, 140, 142(s,1C, C_3 of pyrazole ring), 156(s,1C, C_9 of Benzofuran ring), 153(s,1C,C_6 of quinoline),161(s, 1C,amide linkage), 165(s, 1C, C_5 of 5-oxo-imidazoline ring), 172(s, 1C, C_2 of quinoline ring to which phenyloxy group attached)GC-MS (*m/z)*:719.70 [M], Elemental Anal.Calcd for$C_{45}H_{32}N_6O_4$ calculated;C, 74.99; H, 4.47; N, 11.66; found C, 74.80; H, 4.30; N, 11.40.

5-(Benzofuran-2-yl)-*N*-(4-((6-bromo-2-(*p*-tolyloxy)quinolin-3-yl)methylene)-5-oxo-2-phenyl-4,5-dihydro imidazol-1-yl)-1-phenyl-1*H*-pyrazole-3-carboxamide (5d): IR(KBr, υ_{max} in cm^{-1}): 3403, 3193(N-H str., -CONH-), 3066(C-H str., arom.), 2950(C-H asym. str., aliph.), 2848(C-H sym. str., aliph.), 1457(C-H asym.def., aliph.), 1373(C-H sym.def., aliph.), 1506, 1459(C=C str., arom.), 1072, 1023, 1006(C-H i.p.def., arom.), 833(C-H o.o.p.def., arom.), 1254, 1238(C-O-C asym. str., ether), 1072, 1023(C-O-C sym. str., ether), 1529(C=N str., pyrazole ,imidazole and quinoline nucleus), 1652(C=O str., 5-oxo-imidazolines ring), 1623(C=O str., amide group), 1166(C-N-C str.) , 1072(C-N str.). ^{1}H NMR (DMSO-d_6, 400 MHz):δ (ppm)2.41(s, 3H, -C<u>H</u>$_3$ attached to aromatic ring), 12.16(s,1H, -CON<u>H</u>- linkage), 6.61-8.00(m,25H, aryl ,pyrazole, quinoline ring and ethylenic protons), ^{13}C NMR (DMSO-d_6): δ (ppm)24(-CH_3), 109, 116, 117, 121, 122, 125, 126, 127, 129 ,130, 132, 133, 135, 139, 140, 145(s,1C, C_3 of pyrazole ring), 155(s,1C, C_9 of Benzofuran ring), 153 (s,1C,C_6 of quinoline),160(s, 1C,amide linkage), 168(s, 1C, C_5 of 5-oxo-imidazoline ring), 171(s, 1C, C_2 of quinoline ring to which phenyloxy group attached)GC-MS (*m/z)*:784 [M], Elemental Anal.Calcd for$C_{44}H_{29}BrN_6O_4$ calculatedC, 67.27; H, 3.72; N, 10.70; found C, 67.10; H, 3.56; N, 10.10.

5-(benzofuran-2-yl)-N-(4-((6-methoxy-2-(p-tolyloxy)quinolin-3-yl)methylene)-5-oxo-2-phenyl-4,5-dihydro imidazol-1-yl)-1-phenyl-1H-pyrazole-3-carboxamide (5e):IR(KBr, υmax in cm-1): 3400, 3196 (N-H str., -CONH-), 3061(C-H str., arom.), 2952(C-H asym. str., aliph.), 2843(C-H sym. str., aliph.), 1450(C-H asym.def., aliph.), 1369(C-H sym.def., aliph.), 1504, 1450(C=C str., arom.), 1072, 1029, 1003 (C-H i.p.def., arom.), 830(C-H o.o.p.def., arom.), 1256, 1237(C-O-C asym. str., ether), 1072, 1029(C-O-C sym. str., ether), 1523(C=N str., Pyrazole, imidazole and quinoline nucleus), 1656(C=O str.,5-oxo-imidazolines ring),

1616(C=O str., amide group) , 1164(C-N-C str.), 1072 (C-N str.). 1H NMR (DMSO-d6, 400 MHz):δ (ppm) 2.41(s, 3H, -CH3 attached to aromatic ring), 3.82(s,3H, -OCH3 attached to quinoline ring), 12.20(b,1H, -CONH- linkage), 6.57-8.90(m,25H, aryl ,pyrazole, quinoline ring and ethylenic protons) 13C NMR (DMSO-d6): δ (ppm) 21(-CH3), 55(-OCH3), 106.51, 111, 119, 122, 123, 125, 126, 127, 128 ,129, 132, 133, 135, 139, 140, 144(s,1C, C3 of pyrazole ring), 153(s,1C, C9 of Benzofuran ring), 155 (s,1C,C6 of quinoline),158,161(s, 1C,amide linkage), 168(s, 1C, C5 of 5-oxo-imidazoline ring), 172(s, 1C, C2 of quinoline ring to which phenyloxy group attached) GC-MS (m/z): 736 [M], Elemental Anal. Calcd for C45H32N6O5 calculated; C, 73.36; H, 4.38; N, 11.41; found C, 73.70; H, 4.30; N, 11.24.

General procedure for the determination of zone of inhibition by agar disc-diffusion method: *In vitro* antibacterial activity was determined by using Mueller Hinton Agar obtained from Hi media Ltd., Mumbai. Petri plates were prepared by pouring 10mL of Mueller Hinton Agar for bacteria containing microbial culture was allowed to solidify. Test solutions were prepared with known weight of compound in DMSO and half diluted suitably to give the resultant concentration of 31-1000µg/mL. Whatmann no.1 sterile filter paper discs (6 mm) were impregnated with solution and allowed to dry at room temperature. The discs were then applied and the plates were incubated at 37°C for 24h (bacteria) and the inhibition zone was measured as diameter in four directions and expressed as mean. The results of the antimicrobial screening are illustrated in the Table **2** and **3**.

Table 1: Physical data of the synthesized compound 5a-e

Entry	R	Colour	Recry. solvent	M.F	M.pt. ^{0}C	% yield	Rf
5a	8-CH_3	Yellow	Ethanol	$C_{45}H_{32}N_6O_4$	210	80	0.75
5b	7-CH_3	Yellow	Ethanol	$C_{45}H_{32}N_6O_4$	209	78	0.68
5c	6-CH_3	Yellow	Ethanol	$C_{45}H_{32}N_6O_4$	214	82	0.67
5d	6-Br	Yellow	Ethanol	$C_{44}H_{29}BrN_6O_4$	212	80	0.69
5e	7-Cl	yellow	Ethanol	$C_{44}H_{29}ClN_6O_4$	210	79	0.72

Table 2: Antibacterial activity of 5a-e

Compd. Code	Zone of Inhibition (mm)											
	Gram +ve *S. aureus*						Gram –ve *P.vulgaris*					
	Conc. (µg/mL)						Conc. (µg/mL)					
	1000	**500**	**250**	**125**	**63.5**	**31**	**1000**	**500**	**250**	**125**	**63.5**	**31**
5a	**26**	**24**	20	18	16	**18**	**27**	**24**	**25**	**20**	**19**	**15**
5b	24	**24**	19	15	**17**	15	**26**	22	**24**	18	**17**	**16**
5c	**25**	21	19	16	15	14	25	23	19	20	15	14
5d	**25**	**23**	**22**	**20**	16	**15**	**26**	**25**	19	17	16	**17**
5e	22	**24**	**21**	**19**	**18**	13	27	23	22	**20**	**18**	**16**
DMSO	-	-	-	-	-	-	-	-	-	-	-	-
Std. Drug Chloramphenicol	**25**	**22**	**20**	**19**	**17**	**15**	**26**	**24**	**23**	**21**	**17**	**15**

Table 3: Antibacterial activity of 5a-e

Compd. Code	Zone of Inhibition (mm)											
	Gram –ve											
	E. coli						*S.typhi*					
	Conc. (µg/mL)						Conc. (µg/mL)					
	1000	**500**	**250**	**125**	**63.5**	**31**	**1000**	**500**	**250**	**125**	**63.5**	**31**
5a	**27**	23	**24**	21	16	**14**	**19**	**16**	**13**	10	**10**	**09**
5b	25	22	**24**	**21**	**18**	12	**18**	**17**	**12**	**13**	**09**	**08**
5c	**26**	23	22	17	16	13	15	14	**13**	**12**	08	07
5d	24	**25**	**23**	**22**	**17**	11	18	13	11	**12**	**10**	**08**
5e	**27**	23	23	**21**	14	12	17	**15**	12	**13**	**09**	06
DMSO	-	-	-	-	-	-	-	-	-	-	-	-
Std. Drug Chloramphenicol	**26**	**24**	**23**	**21**	**17**	**14**	**17**	**15**	**12**	**11**	**09**	**08**

RESULTS AND DISCUSSION

The synthesis of the title compound **5a-e** is described in the reaction schemes **1** and **2**. At every stage reaction was monitored with TLC technique. The identities of synthesized compounds have been confirmed using elemental and different spectroscopic techniques such as IR, ^{1}HNMR and ^{13}CNMR and they were also evaluated for their antimicrobial activity. The synthesis of the starting compound 4-((2-(*p*-tolyloxy)-substituted quinolin-3-yl) methylene)-2-phenyloxazol-5(4*H*)-one **3a-e**was achieved in quantitative yields by reacting benzoyl glycine[31] **(1)** with2-(*p*-tolyloxy)-substituted quinoline-3-carbaldehyde **2a-e.** IR spectrum of **3a**showed characteristic absorption bands at 1765, 1796 cm^{-1} due to CO stretch, and another band at1656 cm^{-1} is due to C=N stretch in oxazolone ring. Other absorption band was observed at 1053, 1022 cm^{-1}dueto their C-O-C symmetric stretching.^{1}H NMR of **3a** showed a singlet at δ 2.34 ppm due to three protons of -CH_3 attached to aromatic ring similarly another singlet observed at δ 2.37 ppm was due to three protons of -CH_3 attached to quinoline ring. Aromatic and quinoline ring proton shows multiplet in the range of δ6.57-8.90 ppm, thus all above spectral data confirms the formation of compound **3a.**

The reaction of 5-(benzofuran-2-yl)-1-phenyl-1*H*-pyrazole-3-carbohydrazide **4** with 4-((2-(*p*-tolyloxy)-substituted quinolin-3-yl)methylene)-2-phenyloxazol-5(4*H*)-one **3a-i** in acetic acid solvent afforded **5a-e** in good yields**.** IR spectrum of **5e**showed a distinct absorption band at 3400 cm^{-1} due to NH stretch band while a band at 1523 cm^{-1} aroused due to C=N stretch in pyrazole, band at 1164 cm^{-1} was due to C-N-C stretch. Two characteristic stretching bands due to two carbonyl groups were seen at 1650 cm^{-1} and 1616 cm^{-1}, hence it is confirmed that 4-((2-(p-tolyloxy)-6-methoxy quinolin-3-yl)methylene)-2-phenyloxazol-5(4*H*)-one has been condensed with 5-(benzofuran-2-yl)-1-phenyl-1*H*-pyrazole-3-carbohydrazide (**4**). ^{13}CNMR spectra of compound **5e** also showed a singlet at 168 ppm due to the carbon of (–CONH-), another singlet at 172 ppm was observed due to the carbon of carbonyl group (-CO) similarly, a signal at 55.41 ppm was obtained due to the carbon of methoxy group (-OCH_3). Molecular ion peak for compound**5e** $[M]^{+}$at 736 as obtained in GC-MS spectra and its elemental analysis reveals that % of C, H and N are 73.30, 4.32, 11.39 respectively, is in good agreement with the proposed molecular formula of compound **5e** is $C_{45}H_{32}N_6O_5$.

Antibacterial activity

The antibacterial activity of the synthesized molecule against bacterial strains of Gram positive and Gram negative express in terms of zone of inhibition result depicted in table no **2** and **3.** Antibacterial screening results revealed that most of the synthesised 5-oxo-imidazole derivative **5a-e**exhibit significant antibacterial activity. Test compounds **5a, 5b,** at a conc. 1000µg/mL and 500µg/mL and **5d** and **5e** at a conc. 31µg/mL and 63.5 µg/mL exhibited excellent activity than the standard drugChloramphenicolagainst Gram positive bacteria *S. aureus* and Gram negative bacteria*P. vulgaris, E. coli.* Test compounds **5a, 5b,** at a conc. 31µg/mL and 63.5µg/mL also shows enormous activity against Gram negative bacteria*S.typhi* in compared with reference standard drug at particular concentration. Results also indicated that few of the titled compounds **5c, 5f**showed moderate to good activity at some concentration while the entire synthesizedcompound **5c-e** showed poor activity against Gram negative bacteria*S.typhi.*From the consequences it can established that tested compounds showed variable toxicity against selected strains of bacteria. This incongruity in toxicity it may be due to different substitution on *p*-tolyloxy quinoline which is attached to the basic 5-oxo-imidazole nucleus which enhances the biological activities.

CONCLUSION

we have described the synthesis and antimicrobial screening of series of novel 4-((2-(*P*-Tolyloxy)-substituted quinolin-3-yl)methylene)-4,5-dihydro-5-oxo-2-phenylimidazol-1-yl)-5-(benzofuran-2-yl)-1-phenyl-1*H*-pyrazole-3-carboxamide **5a-e**derivatives through intermediate compound 4-((2-(*P* -Tolyloxy)-substituted quinolin-3-yl) methylene)-2-phenyloxazol-5(4*H*)-one **3a-e**derivatives. Structures of newly synthesised compound **5a-e,** and **3a-e** their purity was checked by physical, analytical and spectral data. The result of bioassay showed that test compounds **5a, 5b, 5d** and **5e** atshowedremarkable activity against Gram positive bacteria *S. aureus* and Gram negative bacteria *P. vulgaris*, *E. coli* andout of all synthesised only test compounds **5a, 5b** showedexcellent activity againstGram negative bacteria*S.typhi* as compared with standard drug reference drug.

ACKNOWLEDGEMENTS

The authors are thankful to the Principal, Government Science College, Gadchiroli, for his support and cooperation. The authors are also gratified to Dr. Roshan Nasare, Dr. Mandar Paingankar, for assisting during antimicrobial activity, similarly the authors are also obliged to, The Director, SAIF, Punjab University, and Chandigarh for providing CHN analysis, FTIR, ^{1}HNMR, ^{13}C NMR and Mass Spectra.

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