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Research Article

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## Synthesis and Antimicrobial Evaluation of Some Novel Chalcones and Pyrazoles from Cyclic Imides under microwave irradiation

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### ABSTRACT

A novel class of chalcones and pyrazoles has been prepared by using neutral alumina; under microwave activation and solvent free reaction condition. The compounds were screened for antimicrobial activity against bacterial strains gram positive bacteria *Staphylococcus aureus*, *Bacillus subtilis* and gram negative bacteria *Escherichia coli*, *Pseudomonas aeruginosa* and two fungal strains *Candida albicans* and *Aspergillus niger*. Some of the compounds exhibited significant inhibitory activity against the microbial strains.

**Keywords:** Chalcones, Pyrazoles, Cyclic imides, Antimicrobial activities

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### 1. Introduction

Heterocycles with nitro [1] moiety offers the great fortune for the designing of novel and compelling medicinal drugs. Heterocyclic compounds such as succinimides [2,3], glutarimides [4], malononitriles [5-8], chalcone [9-12]

centred pyrazoles [13-15] and pyrimidines [16-19] derivatives furnish a very important lead role in the synthesis of organic compounds. The defensive antimicrobial activities [20-22] have seen in the chalcones

and cyclic imides. Natural and synthetic chalcones have shown versatile biological activities such as anti-inflammatory [23], antiviral [24], anticonvulsant [25] antimalarial [26], muscle relaxant [27], antifungal [28], analgesic [29] anti-bacterial [30], anxiety and depression [31], anti-oxidant [32, 33] and anticancer [34,35]. They have also shown expressive cytotoxic activities in contrast to tumour genesis [36] breast cancer, bovine lens [37] aldose reductase [38].

Pyrazole plays very important role in the field of medicinal chemistry. Perhaps, they have been shown significant biological activities like antimicrobial, antineoplastic, anti-inflammatory [39-42] antiviral, antipsychotic, analgesic and antifungal agents so that the pyrazole fragment have been widely used as therapeutic drugs. Also many of them have been involved in vitally important processes, inhibitors of various types of kinases, as receptors antagonists. Rather they can be used for complicated forms of arthritis, and metamizole which is well known analgesic. It is well known that incubation of fluorine atoms into molecules of heterocyclic compounds leads to a significant increase in their biological activities. Thus, pyrazoles proved to be an important “building - blocks” for the synthesis of medicinal drugs and agro-chemicals [43, 44].

## 2. Experimental

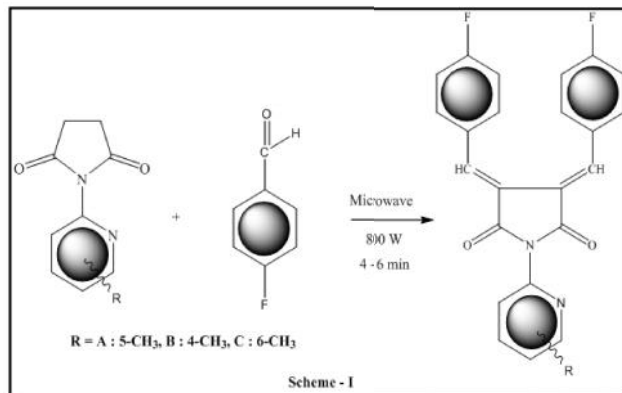
Melting points were recorded in open glass capillaries and were uncorrected. The chemical structures of the obtained compounds were confirmed by spectral analyses. IR spectra in KBr pallets were obtained on Shimadzu and ATR Brucker alpha FT-IR spectrophotometer. <sup>1</sup>H NMR spectra were obtained on and 500.13 MHz by Brucker spectrophotometer. The chemical shifts were reported as parts per million (ppm) with (CH<sub>3</sub>)<sub>4</sub>Si (TMS) as an internal standard. Signal multiplicities are represented by: s (singlet), d (doublet), t (triplet), m (multiplet).

The purity of compound was checked by thin layer chromatography which was performed by using pre-coated silica gel aluminium plates with mixture of diethyl ether and ethyl acetate 7:3 proportion. Anti-microbial and Anti-fungal activities were carried out by Agar diffusion assay (Disk diffusion method, Disk size 6mm) [45,46]. All the compounds (8a-f and 11a-f) were synthesized from the corresponding Succinic and Glutaric Anhydride derivatives and commercially purchased p-fluorobenzaldehyde, neutral alumina (Al<sub>2</sub>O<sub>3</sub>) and ethanol.

### General Procedure of Synthesis:

#### Preparation of Chalcone:

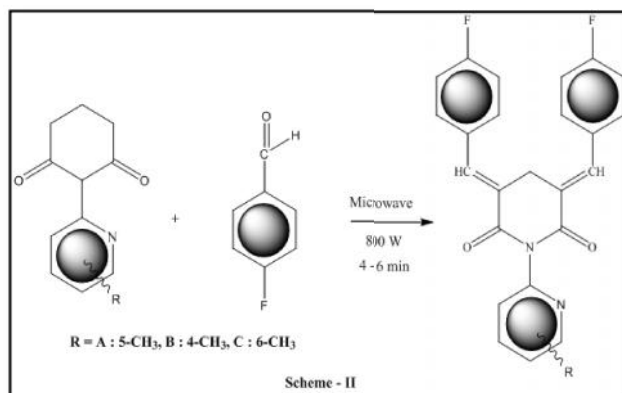
**3,4-bis((E)-4-fluoro benzylidene)-1-(N-methylpyridin-2-yl) pyrrolidine-2, 5-dione (8a-c):** The bis-chalcones (8a-c) derivatives were synthesized by the mixture of 0.01 moles of N-phenyl succinimides and 0.02 mole of p-fluoro benzaldehyde in 1 gm of neutral Al<sub>2</sub>O<sub>3</sub> with the help of microwave irradiations. This mixture is maintained in microwave at 800W power for 4-6 minutes in solvent free condition. The novel developed compounds were recrystallized from ethanol (Scheme – I).



#### Preparation of Chalcone:

#### **3, 5-bis ((E)-4-fluoro benzylidene)-1-(N-methylpyridin-2-yl) piperidine-2,6-dione (8d-f):**

The bis-chalcones (8d-f) derivatives were synthesized by the mixture of 0.01 moles of N-phenyl glutaramides and 0.02 mole of p-fluorobenzaldehyde in 1 gm of neutral Al<sub>2</sub>O<sub>3</sub> with the help of microwave irradiations. This mixture is maintained in microwave at 800W power for 5-8 minutes in solvent free condition. The novel developed compounds were recrystallized from ethanol (Scheme – II).



#### **Physicochemical and analytical data for compounds 8a-f: 3, 4-bis ((E)-4-fluorobenzylidene)-1-(5-methylpyridin-2-yl) pyrrolidine-2, 5-dione (8a)**

Zinc Yellow solid, Yield (85.53%), M. P. 280-82 °C, M.F. C<sub>24</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>F<sub>2</sub> M.W.402.39, Composition: C(71.64%) H (4.01%) F (9.44%) N (6.96%) O(7.95%); IR (KBr): 1176,1685,1720,3197,3127,790,2799,1196,1327,1570,1397 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.44 (m,4H, Ar-H,J=7.5), 7.75 (m,4H,Ar-H,J=7.5), 7.89 (s, 2H, ethylene), 7.96 (d, 1H, pyridine), 7.48 (t, 1H, pyridine, J=7.5), 7.38 (d, 1H, pyridine),2.18 (s, 3H, CH<sub>3</sub>-pyridine).

#### **3, 4-bis ((E)-4-fluorobenzylidene)-1-(4-methylpyridin-2-yl) pyrrolidine-2, 5-dione (8b)**

Melon Yellow solid, Yield (83.24%), M. P. 284-86 °C, M.F. C<sub>24</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>F<sub>2</sub> M.W.402.39, Composition: C(71.64%) H (4.01%) F (9.44%) N(6.96%) O (7.95%); IR (KBr): 1172, 1678,1728,3084,3010,800,2789, 1211, 1307, 1487, 1678, 1425 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.42 (m,4H, Ar-H,J=7.5), 7.72 (m,4H,Ar-H,J=7.5), 7.83 (s, 2H, ethylene), 7.90 (d, 1H, pyridine), 7.36 (d, 1H, pyridine), 7.21 (t, 1H, pyridine),2.28 (s, 3H, CH<sub>3</sub>-pyridine).

**3,4-bis((E)-4-fluorobenzylidene)-1-(6-methylpyridin-2-yl) pyrrolidine-2,5-dione (8e):** Reseda Green solid, Yield (91.10%), M. P. 184-86 °C, M.F. C<sub>24</sub>H<sub>16</sub>O<sub>2</sub>N<sub>2</sub>F<sub>2</sub> M.W. 402.39, Composition: C(71.64%) H(4.01%) F (9.44%) N (6.96%) O(7.95%); IR (KBr): 1167,1672, 1732, 3089,3015,810,2779,1217,1317,1557,1440,1370 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.40 (m,4H, Ar-H,J=7.5), 7.76 (m,4H,Ar-H,J=7.5), 7.85 (s, 2H, ethylene), 6.86 (t, 1H, pyridine,J=7.5), 7.61 (t, 1H, pyridine,J=7.5), 7.31 (t, 1H, pyridine,J=7.5),2.49 (s, 3H, CH<sub>3</sub>-pyridine).

**3, 5-bis ((E)-4-fluorobenzylidene)-1-(5-methylpyridin-2-yl) piperidine-2,6-dione (8d):**

Ivory solid, Yield (89.51%), M. P. 246-48 °C, M.F. C<sub>25</sub>H<sub>18</sub>O<sub>2</sub>N<sub>2</sub>F<sub>2</sub> M.W.416.41, Composition: C(72.11%) H (4.36%) F (9.12%) N (6.76%) O(7.68%); IR (KBr): 1165,1670,1701, 3190,3107,792, 2979, 2935, 1193, 1301, 1575,1402,1446,1389, 1460cm<sup>-1</sup>. <sup>1</sup>HNMR (500.13 MHz, DMSO, ppm): 7.42 (m,4H, Ar-H,J=7.5), 7.73 (m,4H,Ar-H,J=7.5), 7.30 (d, 2H, ethylene),2.61 (t, 2H, methylene), 7.99 (d, 1H, pyridine), 7.53 (t, 1H, pyridine,J=7.5), 7.44 (d, 1H, pyridine),2.20 (s, 3H, CH<sub>3</sub>-pyridine).

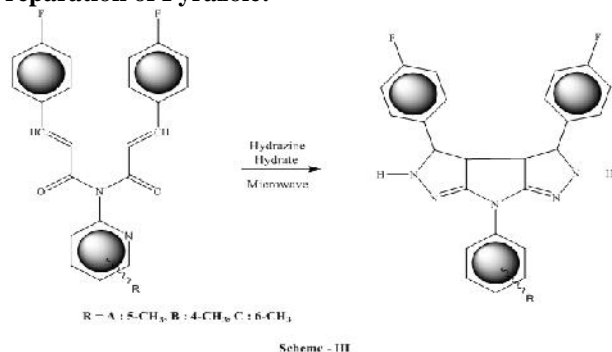
**3,5-bis((E)-4-fluorobenzylidene)-1-(4-methylpyridin-2-yl) piperidine-2,6-dione (8e)**

Khaki Grey solid, Yield (80.58%), M. P. 304-06 °C, M.F. C<sub>25</sub>H<sub>18</sub>O<sub>2</sub>N<sub>2</sub>F<sub>2</sub> M.W.416.41, Composition: C (72.11%) H (4.36%) F(9.12%) N (6.76%) O (7.68%); IR (KBr): 1161, 1572,1740,3010, 818, 2969, 2946, 1223,1297, 1572, 1508, 1369, 1420 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.40 (m,4H, Ar-H,J=7.5), 7.74 (m,4H,Ar-H,J=7.5), 7.32 (d, 2H, ethylene), 2.63 (t, 2H, methylene), 7.94 (d, 1H, pyridine), 7.26 (t, 1H, pyridine), 7.42 (d, 1H, pyridine), 2.30 (s, 3H, CH<sub>3</sub>-pyridine).

**3, 5-bis ((E)-4-fluorobenzylidene)-1-(6-methylpyridin-2-yl) piperidine-2,6-dione (8f)**

Ochre Brown solid, Yield (87.30%), M. P. 218-20 °C, M.F. C<sub>25</sub>H<sub>18</sub>O<sub>2</sub>N<sub>2</sub>F<sub>2</sub> M.W.416.41, Composition: C(72.11%) H (4.36%) F (9.12%) N (6.76%) O (7.68%); IR (KBr): 1157, 1664, 1744, 3040, 2997,815,2959,2950,1230,1311,153, 1427, 1379,1432 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.38 (m,4H, Ar-H, J=7.5), 7.75 (m,4H, Ar-H, J=7.5), 7.34 (d, 2H, ethylene),2.62 (t, 2H, methylene), 6.89 (t, 1H, pyridine, J=7.5), 7.64 (t, 1H, pyridine, J=7.5), 7.36 (t, 1H, pyridine, J=7.5),2.51 (s, 3H, CH<sub>3</sub>-pyridine).

#### Preparation of Pyrazole:

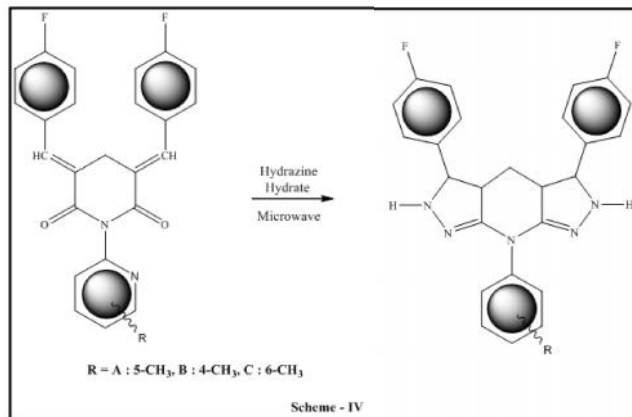


**3,4-bis(4-fluorophenyl)-7-(N-methylpyridin-2-yl)-3, 3a, 3b,4,5, 7-hexahydro-2 H-pyrrolo[2,3-c:5,4-c'] dipyrazole (11a-c):** The previously prepared bis-chalcones (8a-c) of N-phenyl succinimide were used for the preparation of

novel pyrazole derivatives by microwave synthesis. The novel developed compounds were recrystallized from ethanol (Scheme– III).

**Preparation of Pyrazole: 3,5-bis(4-fluorophenyl)-8-(5-methyl pyridin-2-yl)-2, 3, 3a, 4, 4a, 5, 6, 8-octahydro di-pyrazolo [3,4-b:4',3'-e] pyridine (11d-f):**

The previously prepared bis-chalcones (8d-f) of N-phenyl glutarimide were used for the preparation of novel pyrazole derivatives by microwave synthesis. The novel developed compounds were recrystallized from ethanol (Scheme– IV).



**Physicochemical and analytical data for compounds 11a-f: 3, 4-bis (4-fluorophenyl)-7-(5-methylpyridin-2-yl)-3, 3a, 3b, 4, 5,7-hexahydro-2H-pyrrolo[2,3-c:5,4-c'] dipyrazole (11a)**

Saffron Yellow solid, Yield (87.68%), M. P. 314-16 °C, M.F. C<sub>24</sub>H<sub>20</sub>N<sub>6</sub>F<sub>2</sub> M.W.430.45, Composition: C(66.97%) H (4.68%) F (8.83%) N(19.52%); IR (KBr): 1175, 3415, 3032, 838, 2947,1220,1290,1610,1564,1445,1370 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.21 (m,4H, Ar-H,J=7.5),7.25 (m,4H, Ar-H,J=7.5),2.24 (t,2H, methine, J=7.0), 3.6 (d,2H,methine,J=7.0),9.87 (s, 2H, N-H), 7.90 (d, 1H, pyridine), 7.32 (t, 1H, pyridine,J=7.5), 6.64 (d, 1H, pyridine,J=7.5),2.26 (s, 3H, CH<sub>3</sub>-pyridine).

**3,4-bis(4-fluorophenyl)-7-(4-methylpyridin-2-yl)-3, 3a, 3b,4,5,7-hexahydro-2H-pyrrolo[2,3-c:5,4-c'] dipyrazole (11b):**

Yellow Orange solid, Yield (77.52%), M. P. 308-10 °C, M.F. C<sub>24</sub>H<sub>20</sub>N<sub>6</sub>F<sub>2</sub> M.W.430.45, Composition: C(66.97%) H(4.68%) F (8.83%) N(19.52%); IR (KBr): 1169, 3262, 3026, 815, 2927, 1224, 1297, 1616, 1572, 1461, 1331 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.23 (m,4H, Ar-H,J=7.5),7.26 (m,4H, Ar-H,J=7.5), 2.26 (t,2H, methine, J=7.0), 3.8 (d,2H,methine,J=7.0),9.88 (s, 2H, N-H), 7.86 (d, 1H, pyridine,J=7.5), 6.77 (t, 1H, pyridine, J=7.5), 6.62 (d, 1H, pyridine),2.32 (s, 3H, CH<sub>3</sub>-pyridine).

**3,4-bis(4-fluorophenyl)-7-(6-methyl pyridin-2-yl)-3, 3a, 3b, 4,5,7-hexahydro-2H-pyrrolo[2,3-c:5,4-c'] dipyrazole (11c)**

Sulfur Yellow solid, Yield (74.41%), M. P. 288-90 °C, M.F. C<sub>24</sub>H<sub>20</sub>N<sub>6</sub>F<sub>2</sub> M.W.430.45, Composition: C(66.97%) H (4.68%) F (8.83%) N(19.52%); IR (KBr): 1153, 3452,3014,826,2967,1222,1293,1628,1599,1448,1368 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.24 (m,4H, Ar-H,J=7.5),7.27 (m,4H, Ar-H,J=7.5), 2.28 (t,2H, methine, J=7.0), 3.7 (d,2H,methine,J=7.0),9.93 (s, 2H, N-H), 6.31 (t,

1H, pyridine, J=7.5), 7.43 (t, 1H, pyridine, J=7.5), 6.57 (d, 1H, pyridine, J=7.5), 2.56 (s, 3H, CH<sub>3</sub>-pyridine).

**3,5-bis(4-fluorophenyl)-8-(5-methylpyridin-2-yl)-2, 3, 3a, 4, 4a, 5, 6, 8-octahydrodipyrzolo[3,4-b:4',3'-e]pyridine (11d)**

Ivory solid, Yield (87.82%), M. P. 298-96 °C, M.F. C<sub>25</sub>H<sub>22</sub>N<sub>6</sub>F<sub>2</sub> M.W.444.47, Composition: C(67.55%) H (4.99%) F (8.55%) N(18.91%); IR (KBr): 1154, 3449, 3016, 2970, 1154, 1223, 1645, 1547, 1509, 1426, 1368, 1509 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.20 (m, 4H, Ar-H, J=7.5), 7.28 (m, 4H, Ar-H, J=7.5), 2.30 (t, 2H, methine, J=7.0), 3.90 (d, 2H, methine, J=7.0), 1.71 (m, 2H, methylene), 9.88 (s, 2H, N-H), 7.91 (d, 1H, pyridine), 7.33 (t, 1H, pyridine, J=7.5), 6.56 (d, 1H, pyridine, J=7.5), 2.18 (s, 3H, CH<sub>3</sub>-pyridine).

**3,5-bis(4-fluorophenyl)-8-(4-methylpyridin-2-yl)-2, 3, 3a, 4, 4a, 5, 6, 8-octahydrodipyrzolo[3,4-b:4',3'-e]pyridine (11e)**

Olive Brown solid, Yield (85.83%), M. P. 314-16 °C, M.F. C<sub>25</sub>H<sub>22</sub>N<sub>6</sub>F<sub>2</sub> M.W.444.47, Composition: C (67.55%) H (4.99%) F (8.55%) N(18.91%); IR (KBr): 1190, 3096, 3032, 2925, 1190, 1654, 1579, 1548, 1406, 1371, 1487 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.22 (m, 4H, Ar-H, J=7.5), 7.29 (m, 4H, Ar-H, J=7.5), 2.26 (t, 2H, methine, J=7.0), 3.60 (d, 2H, methine, J=7.0), 1.72 (m, 2H, methylene), 9.95 (s, 2H, N-H), 7.87 (d, 1H, pyridine, J=7.5), 6.69 (t, 1H, pyridine, J=7.5), 6.54 (d, 1H, pyridine), 2.24 (s, 3H, CH<sub>3</sub>-pyridine).

**3,5-bis(4-fluorophenyl)-8-(6-methylpyridin-2-yl)-2, 3, 3a, 4, 4a, 5, 6, 8-octahydrodipyrzolo[3,4-b:4',3'-e]pyridine (11f):** Broom Yellow solid, Yield (75.59%), M. P. 286-88 °C, M.F. C<sub>25</sub>H<sub>22</sub>N<sub>6</sub>F<sub>2</sub> M.W.444.47, Composition: C(67.55%) H (4.99%) F (8.55%) N(18.91%); IR (KBr): 1156, 3314, 3199, 3015, 2967, 1156, 1222, 1646, 1599, 1455, 1368, 1506 cm<sup>-1</sup>. <sup>1</sup>H NMR (500.13 MHz, DMSO, ppm): 7.22 (m, 4H, Ar-H, J=7.5), 7.30 (m, 4H, Ar-H, J=7.5),

2.28 (t, 2H, methine, J=7.0), 3.70 (d, 2H, methine, J=7.0), 1.73 (m, 2H, methylene), 9.93 (s, 2H, N-H), 6.32 (t, 1H, pyridine, J=7.5), 7.44 (t, 1H, pyridine, J=7.5), 6.49 (d, 1H, pyridine, J=7.5), 2.48 (s, 3H, CH<sub>3</sub>-pyridine).

### 3. Results and Discussion

#### Chemistry:

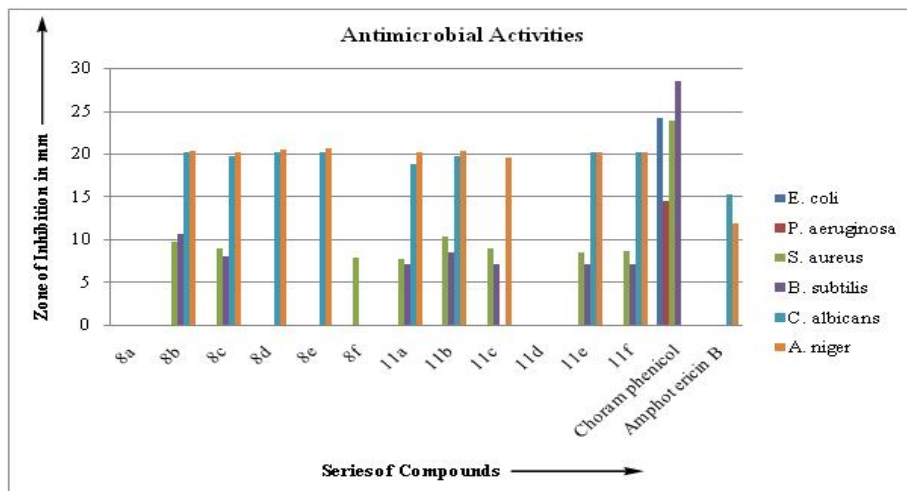
The series of bis-chalcones (8a-f) were synthesized by the reaction between N-phenyl succinimides/N-phenyl succinimides and p-fluro benzaldehyde in presence of neutral Al<sub>2</sub>O<sub>3</sub> with the help of microwave irradiations. The formation of bis-chalcones was confirmed by IR, <sup>13</sup>CNMR and <sup>1</sup>HNMR and elemental analysis. The series pyrazole (11a-f) were prepared by the reaction of bis-chalcones (8a-f) and hydrazine hydrate in the presence of neutral Al<sub>2</sub>O<sub>3</sub> with the help of microwave irradiations and reasonable yield is obtained. The formation of pyrazoles was confirmed by <sup>13</sup>CNMR and <sup>1</sup>HNMR and elemental analysis.

#### Antimicrobial Activities:

All the synthesized bis-chalcones (8a-f) and pyrazole (11a-f) were screened for the anti-bacterial activity against gram positive bacteria *Staphylococcus aureus* (NCIM 2079), *Bacillus subtilis* (NCIM 2250) and gram negative bacteria *Escherichia coli* (NCIM 2109), *Pseudomonas aeruginosa* (NCIM 2036) using DMSO solvent. All these novel synthesized compounds were screened against Fungi (Yeast) *Candida albicans* (NCIM 3471) and *Pergillusniger* (NCIM 545). The bacterial cultures were purchased from NCIM: National Collection of Industrial Microorganisms, National Chemical Laboratory (NCL), Pune 411008 [India]. Some of the compound showed moderate to good activities against gram positive bacteria *S. aureus*, *B. subtilis* and synergetic activities against Fungi *C. albicans*, *A. niger*; as shown in the Table-I and Graph-I;

**Table-I:** Ant-imicrobial activities of bischalcone (8a-f) and Pyrazole (11a-f)

Sr. No.	Sample	<i>E. coli</i>	<i>P. aeruginosa</i>	<i>S. aureus</i>	<i>B. subtilis</i>	<i>C. albicans</i>	<i>A. niger</i>
		Mean±SD	Mean ±SD	Mean ±SD	Mean ±SD	Mean ±SD	Mean ±SD
1	8a	- - -	- - -	- - -	- - -	- - -	- - -
2	8b	- - -	- - -	9.69±0.17	10.55±0.11	20.12±0.11	20.30±0.25
3	8c	- - -	- - -	8.87±0.12	7.96±0.13	19.66 ±0.22	20.20±0.06
4	8d	- - -	- - -	- - -	- - -	20.15± 0.04	20.50±0.05
5	8e	- - -	- - -	- - -	- - -	20.16±0.04	20.60±0.05
6	8f	- - -	- - -	7.70±0.10	- - -	- - -	- - -
7	11a	- - -	- - -	7.69±0.18	7.15±0.06	18.84±0.15	20.20±0.01
8	11b	- - -	- - -	10.28± 0.07	8.39±0.07	19.75±0.19	20.30±0.03
9	11c	- - -	- - -	8.94±0.08	7.12±0.13	- - -	19.60±0.07
10	11d	- - -	- - -	- - -	- - -	- - -	- - -
11	11e	- - -	- - -	8.51±0.07	7.117±0.11	20.13±0.11	20.20±0.06
12	11f	- - -	- - -	8.58±0.07	7.00±0.00	20.14±0.03	20.10±0.03
	<i>Choramphenicol</i>	24.09±0.10	14.39±0.07	23.92±0.17	28.43±0.29	NA	NA
	<i>Amphotericin B</i>	NA	NA	NA	NA	15.21±0.15	11.8±0.08



Graph-I: Antimicrobial activities of bis-chalcone (8a-f) and Pyrazole (11a-f)

#### 4. Conclusion

In the present work, we have focused our attention on the synthesis of a novel class of chalcones and pyrazoles from cyclic imides by using neutral alumina; under microwave activation & solvent free reaction condition. Antimicrobial study of chalcones and pyrazoles demonstrate that they are more active and selective against gram positive bacteria *S. aureus*, *B. subtilis* and Fungi *C. albicans*, *A. niger*; for these reasons, we can indicate chalcones and pyrazoles as a useful scaffolds for medicinal chemistry and we plan to extend and report structure-activity relationship studies, including other substituents, in a further communication.

#### 5. References

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