Microwave assisted synthesis, characterization and antimicrobial activity of novel bipyrazole derivatives

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Abstract

*N'-[-1-phenylethylidene] pyridine-4-carbohydrazide (3a-f) were prepared by reacting with substituted acetophenone and iso-nicotinic acid hydrazide and were reacted with Phosphorous oxy chloride and Dimethyl formamide to yield 3-phenyl-1-(pyridin-4-ylcarbonyl)-1H-pyrazole-4-carbaldehyde (4a-f). Solution of substituted acetophenone and Pyrazole-4-carbaldehyde were reacted to obtain 1-phenyl-3-[3-phenyl-1-(pyridin-4-ylcarbonyl)-1H-pyrazol-4-yl] prop-2-en-1-one (5a-f). To Synthesize 5, 3'-diphenyl-1', 2-diisonicotinoyl-3, 4-dihydro-1'H, 2H-3, 4'-bipyrazole (6a-f), 1-phenyl-3-[3-phenyl-1-(pyridin-4-ylcarbonyl)-1H-pyrazol-4-yl] prop-2-en-1-one was reacted with iso-nicotinic acid hydrazide. Moreover, the compounds revealed antimicrobial activity together with significant antifungal activities to some extent. Compound **6f** with methoxy substitution, was found to be the most potent compound of the series with antifungal activity comparable to the standard drug, Fluconazole, against A. niger. It was followed by compounds **6b** and **6c** which depicted significant activity against C. albicans and A. niger. Other compounds in the current research have shown moderate activity against tested antibacterial and antifungal strains.*

Keywords: Bipyrazole, heterocyclic, antibacterial, antifungal, substituted acetophenone and iso-nicotinic acid hydrazide.

INTRODUCTION

Antifungal and antibacterial activities of Bipyrazole derivatives are most rapidly studied and some of them are in clinical practice as antimicrobial agents. Bipyrazole derivatives are an important class of heterocyclic compounds and many of them are reported to have the broad spectrum of biological activities. In the present study, Bipyrazole derivatives have been undertaken as target heterocyclic nucleus because of their remarkable pharmacological activities reported so far. Bipyrazole derivatives have been reported to possess potential antitumor [1], anti-inflammatory [2-4], antimicrobial [5], cytotoxic [6, 7], antiallergic [8], cardiovascular [9], and diuretic [10] activities. Bipyrazoles were also found to have useful as insecticidal [11], herbicidal [12] and fungicidal [13-15] activity in the photographic and paint industry. Bipyrazole derivatives were also used as new class of supramolecular complexes, organometallic cage-like structures and self-assembling metallomacrocycles with Bipyrazole ligands that are promising as catalysts, molecular mimics, molecular magnetic devices and sensors [16-18]. The Bipyrazole derivatives were applied as efficient ligands in the palladium-catalyzed C-O and C-N cross-coupling reactions of aryl halides with primary alcohols and with urea derivatives, respectively [19-23].

Microwave irradiation in solvent free conditions has well demonstrated its utility as the energy source in many organic reactions including cycloadditions. In this study, we have used this microwave assisted synthesis for synthesizing some newer derivatives of bipyrazole and thereafter evaluating their biological activities over selected microbial strains.

For biological activity evaluation, both antibacterial and antifungal activities have been undertaken.

MATERIALS AND METHODS

Reagents and chemicals

All the substituted aldehydes, acetone and isoniazide were obtained from Hi-media Chem. Ltd. and Lancaster Ltd. The solvents and chemicals were procured from Macleods Pharmaceutical Ltd., Hayman Ltd., Fischer, S.D. Fine Chem Ltd., and Loba Chemie Pvt. Ltd. All the compounds procured were purified and dried using standard methods, before use. Melting points were determined using melting point apparatus MP-DS, TID 2000 and were uncorrected. Purity of the compounds was routinely checked by TLC using plates coated with silica gel-G. Iodine vapour was used as visualizing agent. UV spectra were recorded on JASCO V-530 UV/VIS spectrophotometer in the Department of Pharmaceutical Analysis, Sumandeep Vidyapeeth University, Vadodara, Gujarat. Microwave synthesis was carried out using DAEWOO KOG-370A at Pharmaceutical Chemistry Laboratory, Dr. B. C. Roy College of Pharmacy and Allied Health Sciences, Durgapur. IR spectra were recorded on JASCO FT/IR-140, Department of Pharmaceutical Analysis, Sumandeep Vidyapeeth University, Vadodara, Gujarat. Mass spectra were recorded on LCMS-2010A Mass Spectrometer at Quest Research and Training Institute. PMR spectra were recorded at IIT, Madras and Quest Research and Training Institute.

For antibacterial screening, the following is used:

Media Mueller- Hinton agar

Mueller Hinton broth gelled by the addition of 2% agar (bacteriological grade).

Ingredients

Casein enzymic hydrolysate : 7.4 ± 0.2
Beef infusion : 300gm/ L
Soluble starch : 1.5 gm/L
Final pH at 25°C : 17.5 gm/L

Preparation

The ingredients were dissolved in distilled water with the aid of heat and pH was adjusted to 7.2-7.6 using alkali or dilute acid.

Sterilization

15-20 ml of Mueller Hinton agar was transferred to test tubes and sealed with non-absorbent cotton. It was then autoclaved at a pressure of 15 psi (121°C) for not less than 15 minutes.

Organisms used

Micrococcus NCIM 2079, *Pseudomonas aeruginosa* NCIM 2036, *Escherichia coli* NCIM 2118 and *Bacillus subtilis* NCIM 2063 were procured from National Chemical Laboratory, Pune and stored in the Pharmaceutical Biotechnology Laboratory, Dr. B. C. Roy College of Pharmacy and Allied Health Sciences, Durgapur. The strains were confirmed for their purity and identity by Gram's staining method and their characteristic biochemical reactions. The selected strains were preserved by sub culturing them periodically on nutrient agar slants and storing them under frozen conditions. For the study, fresh 24 hr broth cultures were used after standardization of the culture.

Working conditions

The entire work was done using horizontal laminar flow hood so as to provide aseptic conditions. Before commencement of the work air sampling was carried out using a sterile nutrient agar plate and exposing it to the environment inside the hood. After incubation it was checked for the growth of microorganism and absence of growth confirmed aseptic working conditions.

Preparation of inoculum

The inoculum for the experiment was prepared fresh in Mueller Hinton broth from preserved frozen slants. It was incubated at 37°C for 18-24 hrs and used after standardization.

Compounds used : Bipyrazoline derivatives
Standard used : Ciprofloxacin (10 μg/disc)
Vehicle used : Dimethyl sulphoxide

Furthermore, for antifungal activity screening the following chemicals are used.

Media
Sabouraud Dextrose Agar (composition followed)
Mycological peptone: 10 gm.
Dextrose: 40 gm.
Agar: 15 gm.
Final pH at 25°C : 5.4 ±0.2
Water to make 1000 ml

Preparation

65 gm. of Sabouraud dextrose agar was suspended in 1000 ml. of distilled water and boiled to dissolve the medium completely.

Organism used

Candida albicans NCIM 3102 and *Aspergillus niger* NCIM 596 were procured from National Chemical Laboratory, Pune and stored in the Pharmaceutical Biotechnology Laboratory, Dr. B. C. Roy College of Pharmacy and Allied Health Sciences, Durgapur.

Compounds used : Bipyrazoline derivatives
Standard used : Fluconazole (10 μg /disc)
Vehicle used : Dimethyl sulphoxide

Synthesis of target compounds

The title compounds were prepared in the following steps: [24-26]

General procedure for synthesis of Synthesis of *N'-[-1-phenylethylidene] pyridine-4-carbohydrazide* (**3a-f**)

A solution of isoniazide (0.01 mole) and acetophenone/substituted acetophenone (0.01 mole) in ethanol (15 ml) with a few drops of glacial acetic acid was irradiated under microwave irradiation for 2 to 3 minutes (Scheme 1) The reaction mixture was cooled. The solid that separated on cooling was filtered, washed with cold ethanol, dried and recrystallized from chloroform.

2.2.2 Synthesis of 3-(4'-substituted) phenyl-1-(pyridin-4-ylcarbonyl)-1H-pyrazole-4-carbaldehyde (**4a-f**)

To the Vilsmeir Haack complex, prepared from Dimethyl formamide (10 ml) and Phosphorous oxy chloride (0.012 mole) at 0°C, the carbohydrazide prepared in the above step-1 (0.04 mole) was added and the reaction mixture was subjected to microwave irradiation for 3-4 min. The reaction mixture was cooled and poured into ice-cold water. The product which separated on neutralization with sodium bicarbonate solution was filtered and recrystallized from ethanol-dimethyl formamide.

2.2.3 Synthesis of 1-(4'-substituted)-phenyl-3-[3-phenyl-1-(pyridin-4-ylcarbonyl)-1H-pyrazol-4-yl] prop-2-en-1-one (**5a-f**)

To a solution of same ketone which was used in the step-1 (0.01 mole) and the corresponding Pyrazole-4-carbaldehyde obtained from step-2, (0.02 mole) in dry ethanol (20 ml) taken in a Borosil beaker (100ml), a catalytic quantity of sodium hydroxide (1-2 pellets) was added and the reaction mixture was zapped inside a microwave oven for 30 seconds to 2 minutes (at 210 watts *i.e.* 30% microwave power) and then cooled in a ice bath. The product formed was filtered and washed with ethanol (5ml) followed by water till the washings are neutral. The purity of the compounds was checked by TLC using methanol: water (8:2, v/v) as solvent system.

2.2.4 Synthesis of5, 3'-substituted diphenyl-1', 2-diisonicotinoyl-3, 4-dihydro-1'H, 2H-3, 4'-bipyrazole (**6a-f**)

A mixture of chalcones as prepared in the above step-3 (0.001 mol) and isoniazide (0.001 mol) was zapped inside a domestic microwave oven for 8 to 10 minutes (at 640 watts i.e. 80% microwave power) in presence of piperidine as a catalyst. After cooling, the solution was poured on to crush ice; the

product obtained was filtered and recrystallized from dichloromethane-methanol. The purity of the compounds was checked by TLC using methanol: water (8:2, v/v) as solvent system.

2.2.5 Characterization studies

Melting points were determined in open capillary lubes and are uncorrected. All the chemicals and solvents (ethanol and acetone) used were of laboratory grad and solvent were purified by suitable methods [27]. IR spectra (KBr, cm^{-1}) were recorded on a JASCO FT/IR-410 spectrometer. 1H NMR spectra was recorded on Brucker 300 MHz NMR spectrometer (chemical shifts in δ ppm) using TMS as an internal standard. Mass spectrum was recorded on LCMS-2010A Mass Spectrometer. The purity of the compounds was ascertained by thin layer chromatography on aluminium plates percolated with silica gel G (Merck) in various solvent systems using iodine vapours as detecting agent. Reactions were carried out in a Daewoo KOG-370A domestic microwave oven at 2450 MHz.

2.3 Evaluation of antimicrobial activity by Kirby-Bauer Method

Mueller Hinton agar plates were prepared aseptically to get a thickness of 5-6 mm. The plates were allowed to solidify and inverted to prevent the condensate falling on the agar surface. The plates were dried at 37°C before inoculation. The organism was inoculated in the plates prepared earlier, by dipping a sterile swab in the previously standardized inoculum, removing the excess of inoculum by pressing and rotating the swab firmly against the sides of the culture tube above the level of the liquid and finally streaking the swab all over the surface of the medium 3 times, rotating the plates through an angle of 60° after each application. Finally, the swab was pressed round the edge of the agar surface. It was allowed to dry at room temperature, with the lid closed. The sterile disc containing test drugs, standard and blank were placed on the previously inoculated surface of the Mueller Hinton agar plate and it was kept in the refrigerator for one hour to facilitate uniform diffusion of the drug. Plates were prepared in triplicate and they were then incubated for 18-24 hrs. Observations were made for zone of inhibition around the drugs and compared with that of standard. All the compounds synthesized were tested for antibacterial activity against gram positive and gram-negative bacteria. Saturated solutions of the compounds were first studied for activity and the compounds with zones greater than 15mm were taken for quantitative studies.

2.3 Evaluation of antifungal activity

Sabouraud dextrose agar plates were prepared aseptically to get a thickness of 5-6 mm. The plates were allowed to solidify and inverted to prevent the condensate falling on the agar surface. The plates were dried at 25°C just before inoculation.

The organisms (*Candida albicans* NCIM 3102 and *Aspergillus niger* NCIM 596) were inoculated in the plates prepared earlier by dipping sterile swab in the inoculum, removing the excess of inoculum by pressing and rotating the swab firmly against the sides of the culture tube above the level of the liquid and finally streaking a swab all over the surface of the medium three times, rotating the plates through the angle of 60° after each application. Finally, the swab was pressed round the edges of the agar surface. It was left to dry at room temperature with the lid closed. Sterile discs containing the test, standard and blank were placed in the petridish aseptically. 10 µg / disc of saturated solutions was used.

Plates were prepared in triplicate and they were incubated at 25°C for 24-48 hrs, after placing them in the refrigerator for one hour to facilitate uniform diffusion. Observations were made for the zone of inhibition around the discs and compared with that

of Fluconazole, the standard. All the compounds were tested for antifungal activity.

RESULTS AND DISCUSSION

Synthesis

N'-[-1-phenylethylidene] pyridine-4-carbohydrazide (3a-f) were prepared by following the standard protocol (57) as shown in scheme 1 and were reacted with Phosphorous oxy chloride and Dimethyl formamide to yield 3-phenyl-1-(pyridin-4-ylcarbonyl)-1H-pyrazole-4-carbaldehyde (4a-f). Solution of substituted acetophenone and Pyrazole-4-carbaldehyde were reacted to obtain 1-phenyl-3-[3-phenyl-1-(pyridin-4-yl-carbonyl)-1H-pyrazol-4-yl] prop-2-en-1-one (5a-f). To Synthesis of 5, 3'-diphenyl-1', 2-diisonicotinoyl-3, 4-dihydro-1'H, 2H-3, 4'-bipyrazole (6a-f) were reacted with1-phenyl-3-[3-phenyl-1-(pyridin-4-ylcarbonyl)-1H-pyrazol-4-yl] prop-2-en-1-one and iso-nicotinic acid hydrazide. For synthesizing pyrazole ring, the route of hydrazone formation has been undertaken because of high reactivity of phenyl (substituted) hydrazine with acetophenone. $POCl_3$ based cyclization has been performed followed by subsequent carbonylation by Vilsmayer Haack reaction. The carbonylation has been performed in order to allow free carbonyl groups for further Claisen condensation. After Beavault Blanc reduction with Na and C_2H_5OH, incorporation of further substituted phenyl hydrazine makes it bipyrazole derivative with two substituted phenyl groups. The assigned structure and molecular formula of the newly synthesized compounds (6a-f) were confirmed and supported by ^1H NMR, Mass spectra, and IR data, which was in full agreement with proposed structures.

The compounds were screened in vitro for their antibacterial and antifungal potential by disc diffusion assay against selected pathogenic bacteria and human pathogenic fungi. The Physical data of synthesized compounds are presented in Table 1.

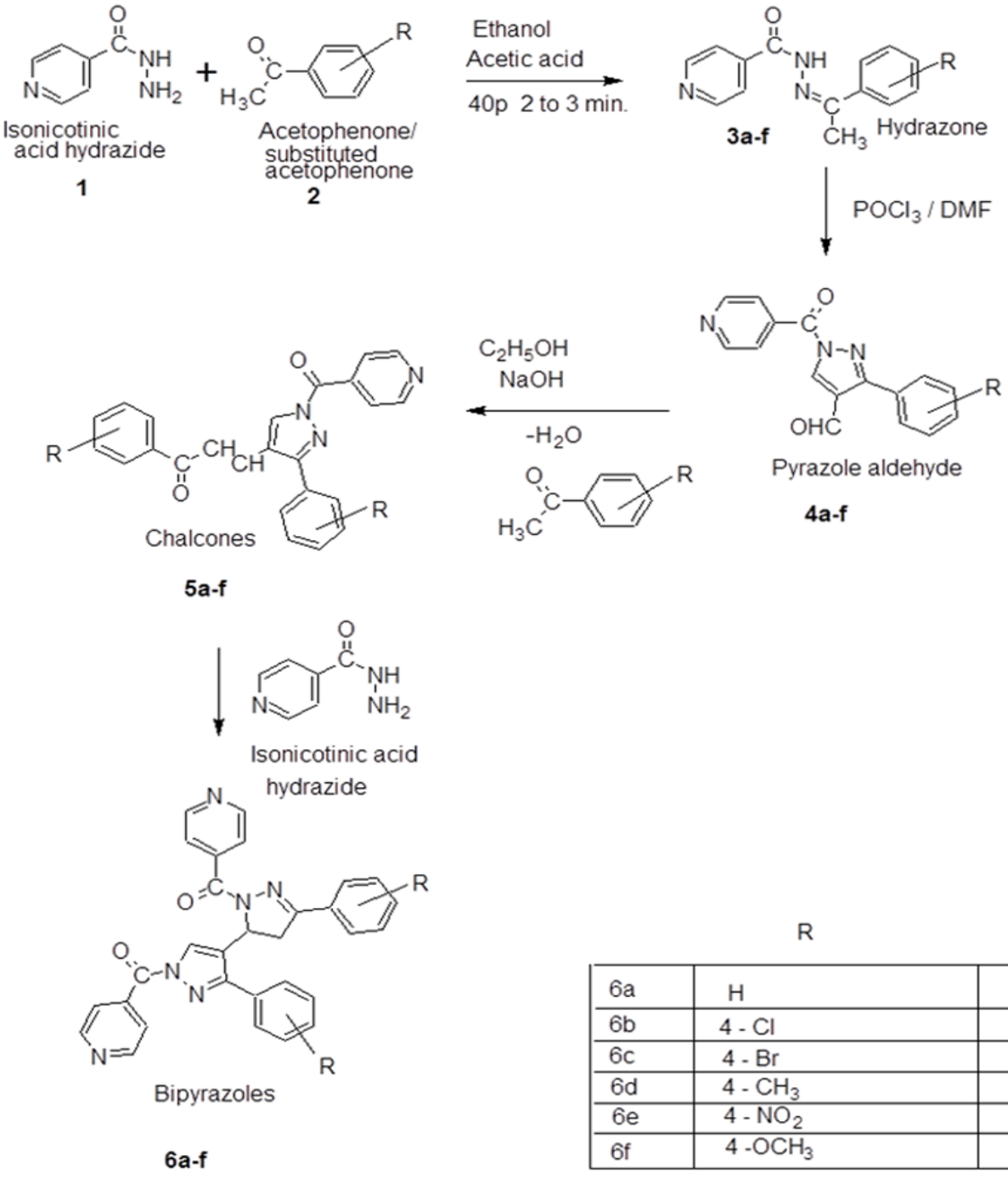
Table 1. Physical and analytical data of the synthesized compounds

Compound Code	R	Molecular Formula	Molecular Weight	Reaction time	% Yield	Melting point	R_f value
6a	H	$C_{30}H_{22}N_6O_2$	499	10 min	73	165 °C	0.7213
6b	4-Cl	$C_{30}H_{20}Cl_2N_6O_2$	567	10 min	80	163 °C	0.6978
6c	4-Br	$C_{30}H_{20}Br_2N_6O_2$	656	10 min	81	232 °C	0.7134
6d	4-CH_3	$C_{32}H_{26}N_6O_2$	527	10 min	76	187 °C	0.6995
6e	4-NO_2	$C_{30}H_{20}N_8O_6$	589	10 min	80	260 °C	0.7251
6f	4-OCH_3	$C_{32}H_{26}N_6O_4$	559	10 min	77	192 °C	0.7358

Biological Activity

The antimicrobial activity was determined using disc diffusion method (30) by measuring the inhibition zone in mm. All the newly synthesized compounds, *i.e.* (6a-f) were screened in vitro for their antibacterial activity against two Gram-positive strains (*Staphylococcus aureus* and *Bacillus subtilis*) and two Gram-negative strains (*Escherichia coli* and *Pseudomonas aeruginosa*) at a concentration of 500 µg/mL. Antifungal activity was tested against *Candida albicans* and *Aspergillus niger* at a concentration of 500 µg/mL. Ciprofloxacin (10 µg/disc) was used as a standard

drug for anti-bacterial screening and Fluconazole (10 µg/disc) was used as a standard drug for antifungal screening.



	R	R
6a	H	H
6b	4 - Cl	4 – Cl
6c	4 - Br	4 – Br
6d	4 - CH_3	4 – CH_3
6e	4 - NO_2	4 – NO_2
6f	4 -OCH_3	4 – OCH_3

Scheme 1. Synthesis of compounds 6(a-f)

All synthesized compounds exhibited moderate anti-bacterial activities and significant antifungal activities. Each experiment was done in triplicate and the average reading was taken. The results of antibacterial and antifungal activity expressed in term of zone of inhibition are reported in Table 2.

Table 2. Antimicrobial activity-sensitivity testing of compounds (6a-f)

Compound No.	Zone of inhibition in mm					
	Antibacterial activity				Antifungal activity	
	S. aureus	*B. subtilis*	*E. coli*	*P. aeruginosa*	*C. albicans*	*A. niger*
6a	10	11	8	9	17	19
6b	13	15	9	8	22	24
6c	10	11	8	9	19	22
6d	12	12	8	9	15	22
6e	13	14	9	8	17	20
6f	09	11	8	8	22	26
Ciprofloxacin	26	26	28	25	-	-
Fluconazole	-	-	-	-	28	26

Structure assignment

Compound 6a:

FT-IR (KBr, cm^{-1}): 3076, 1569, 1460 (Pyridine), 1419, 1348, 1168, 1027 (Pyrazole), 3008 (benzene), 1662 (-C=O stretch); ^{1}H NMR (300 MHz, DMSO-d_6, ppm): δ 2.49 (d, 2H, CH2 of Bipyrazole), 7.5 (t, 1H, CH of Bipyrazole), 7.83 (m, 16H, Ar-H); *m/z* 500 (M+H).

Compound 6b:

FT-IR (KBr, cm^{-1}): 3008, 1594, 1485 (Pyridine), 1431, 1399, 1092 (Pyrazole), -C=O stretch (1649), C-Cl stretch (664); ^{1}H NMR (300 MHz, DMSO-d_6, ppm): δ 2.48 (d, 2H, CH_2 of Bipyrazole), 7.6 (t, 1H, CH of Bipyrazole), 7.8 (m, 16H, Ar-H), *m/z* 569 (M+2H).

Compound 6c:

FT-IR (KBr, cm^{-1}): 3059, 1597, 1486 (Pyridine), 1368, 1177 (Pyrazole), 1649 (-C=O stretch), 582 (C-Br stretch); ^{1}H NMR (300 MHz, DMSO-d_6, ppm): δ 2.52 (d, 2H, CH2 of Bipyrazole), 7.6 (t, 1H, CH of Bipyrazole), 7.82 (m, 16H, Ar-H), *m/z* 657 (M+H).

Compound 6d:

FT-IR (KBr, cm^{-1}): 3032, 1606, 1456 (Pyridine), 1369, 1165, 1092 (Pyrazole), 1666 (-C=O stretch), CH_3 (2986, 2956); ^{1}H NMR (300 MHz, DMSO-d_6, ppm): δ 2.62 (d, 2H, CH2 of Bipyrazole), 7.6 (t,1H, CH of Bipyrazole), 7.8 (m, 16H, Ar-H), *m/z* 527 (M, the molecular ion).

Compound 6e:

FT-IR (KBr, cm^{-1}): 3003, 1519, 1417 (Pyridine), 1519, 1350, 1149, 1092 (Pyrazole), 1649 (-C=O stretch); ^{1}H NMR (300 MHz, DMSO-d_6, ppm): δ 2.52 (2H, d, CH2 of Bipyrazole), 7.5 (t, 1H, CH of Bipyrazole), 7.87 (m,16H, Ar-H). *m/z* 589 (M, the molecular ion).

Compound 6f:

FT-IR (KBr, cm^{-1}): 3039, 1602, 1393 (Pyridine), 1393, 1180, 1027 (Pyrazole), 1644 (-C=O), 2823 (-OCH_3); ^{1}H NMR (300 MHz, DMSO-d_6, ppm): δ 2.49 (d, 2H, CH2 of Bipyrazole), 7.5 (t, 1H, CH of Bipyrazole), 7.87 (m, 16H, Ar-H), 3.73-3.79 (m, 6H, OCH_3 of Bipyrazole). *m/z* 561 (Pseudo molecular ion M+2H).

The corresponding NMR and IR spectra of the compounds have been provided in supplementary information (Fig. S1- S12).

CONCLUSION

Newly synthesized Bipyrazoline derivatives (6a-f) were evaluated for antimicrobial activities. The results of antimicrobial studies possess antibacterial activity to certain extend and significant antifungal activities. Compound 6f with methoxy substitution, was found to be the most potent compound of the

series with antifungal activity to some extent better than that of the standard drug, i.e. Fluconazole, against *A. niger*. It was followed by compounds 6b and 6c which depicted significant action as that of the standard drug against *C. albicans* and *A. niger*. Rests of the compounds have shown moderate activity against tested antibacterial and antifungal strains. Even though, the synthesized Bipyrazoline derivatives did not exhibit appreciable activity, the data reported in this paper maybe helpful guide for the medicinal chemists who are working in this area.

For supplementary information, please go to www.bcrcppharmawave.net, vol 10, 2017

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