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Microwave Assisted Synthesis and Antibacterial Activity of Substituted 2-Furyl Pyrazoline Derivatives

KEYWORDS: Pyrazoline,2-furyl acetone, Spectral data, antibacterial activity.

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ABSTRACT *Some new 3(furan-2-yl)-1,5-diphenyl 4-5dihydro-1-H-pyrazole;5(4-chorophenyl)-3-(furan-2-yl)-1- phenyl 4-5- dihydro -1-H-pyarzole;5(4-nitrophenyl)-3-(furan-2-yl)-1-phenyl 4-5- dihydro-1-H-pyarzole etc.have been synthesized employing microwave technique and confirmed bysuitable spectroscopic technique such as 1H NMR. The compounds were screened for their in vitrobacterial activity against S. aureus E. coli, S. typhi bacteria.*

Introduction:
Pyrazolines are well known and important nitrogen-contaning 5-membered hetrocyclic compound and various methods have been worked out for their synthesis[1].Microwave assisted synthesis ofsome novel 2-pyrazoline derivatives also reported[2].Neumerous pyrazoline derivatives have been found to possess considerable biological activities,which stimulated the research activity in this field.As a result,a large number of pyrazolines using different synthetic methods for their preparation have been described in the chemistry literature.An especially popular procedure is based on the reaction of α-β unsaturated aldehydes or ketones with hydrazines [3-4].Some synthesis of 3(2-furyl) pyrazoline derivative and its studies on antidepressant & anticonvulsant given by ozdemir[5].Pyrazolines can be synthesized by the rection between chalcone & aryl hydrazine using catalytic amount of acetic acid in ethanol as a solvent under reflux condiction [6]and acetic acid as a solvent[7].Simple methods for synthesizing non-convential method using microwave condition which does not need any catalyst [8].

On the other hand,microwave assisted organic reactions have emerged as a new Lead in organic synthesis with important advantages like highly accelerated rate of reaction along with improvement in yield and quality of product[9].

Experimental:
All melting points were determined in open capillary tubes and are uncorrected.All the chemicals and solvents used were of laboratory grade.1H NMR spectra was recorded on Brucker 300MHz,NMR spectrometer using TMS as an internal standard.

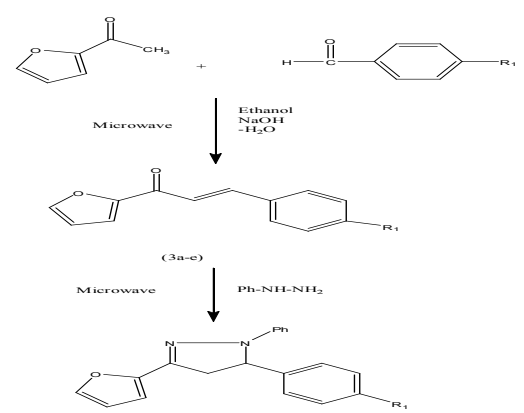
General method:
A solution of 2-furyl acetone(0.05mole) and appropriately substituted benzaldehyde (0.05mole) in ethanol takaen in conical flask.Sodium hydroxide was added into reaction mixture.Reaction mixcture zapped in microwave oven for 30 sec to 1 min at180 watt and then cooled in refrigerator overnight. The product obtained was filtered and washed with water and recrystallization from ethanol.

Then these synthesized chalcones reacts with phenyl hydrazine in microwave oven at 180 watt gives different substituted pyrazolines.

Synthesis of 3-(furan-2-yl)-1,5-diphenyl-4-5-dihydro-1-H-pyrazole(4a-f)
A mixture of substituted chalcone (3a-f)(0.02mole)and phenyl hydrazine(0.02mole) was zapped inside a microwave oven for 1 to 3 min at 180 watt.After cooling ,the solution was poured into crushed ice and the product obtained was filtered and recrystalised using ethanol.

Physical data of synthesized compound are presented as-Table No.1

compound	R1	Reaction time(min)	Mol. formula	Yield	Mol. wt.	m.pt °c
4a	-H	1.51	$C_{19}H_{16}N_2O$	60%	288	175°c
4b	-Cl	2.35	$C_{19}H_{15}N_2OCl$	61.24%	322.5	190 °c
4c	$-CH_3$	1.56	$C_{20}H_{18}N_2O$	50%	302	180 °c
4d	-NO2	1.35	$C_{19}H_{15}N3O_3$	53%	333	210°c
4e	-Br	2.56	$C_{19}H_{15}N_2OBr$	65%	366	185 °c



Scheme 1:-Synthesis of title compound (4a-e)

3-(furan-2-yl)-1,5-diphenyl-4-5-dihydro-1-H-pyrazole (4a)
[1]H NMR 7.75(CH,S,of 2-furan),5.19(CH,t ,of methine),3.90(CH 2,d,methylene),6.83(CH,S,of 1-benzene ,1-N-C),7.40(CH,S,of 1-benzene 1-C-C)

5(4-Chlorophenyl)-3-(furan-2-yl)-1,5-diphenyl-4-5-dihydro-1-H-pyrazole (4b)
[1]H NMR 7.75(CH,S,of 2-furan),5.19(CH,t ,of methine),3.90(CH2,d,methylene),7.44(CH,of-C-Cl),6.83(CH,S,1-benzene,1-N-C)

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3-(furan-2-yl)-1-Phenyl-5$_{-P}$-tolyl-4,5-dihydro-1-H-pyrazole (4c)
^{1}H NMR 7.75(CH,S,of 2-furan),5.19(CH,t ,of methine),3.90(CH2,d,methylene)7.12(CH,of –C-O), 6.83(CH,of 1-benzene,1-N-C).2.35(for –CH_3)

5(4-Nitrophenyl)-3-(furan-2-yl)-1,5-diphenyl-4-5-dihydro-1-H-pyrazole (4d)
^{1}H NMR 7.75(CH,S,of 2-furan),5.19(CH,t ,of methine),3.90(CH2,d,methylene), 6.83(CH,S,1-benzene,1-N-C),8.21(CH,of 1-benzene ,1-N(=o)=o)

5(4-Bromophenyl)-3-(furan-2-yl)-1,5-diphenyl-4-5-dihydro-1-H-pyrazole (4e)
^{1}H NMR 7.75(CH,S,of 2-furan),5.19(CH,t ,of methine),3.90(CH2,d,methylene),7.92(CH,of –C-Br), 6.83(CH,S,1-benzene,1-N-C)

Antimicrobial activities
Antibacterial activity
Staphylococcus aureus was taken as gram positive strain, and Escherichia coli and Salmonella typhi species were taken as gram negative strains; they have been used for the present study.The antimicrobial activity was determined using disc diffusion method[11] by measuring the inhibition zone in mm.All the synthesized compounds exhibited significant antibacterial activity.

Table No.2

Compounds	Antimicrobial Activity (Zone of Inhibition in mm)		
	S.aureus	E.coli	S.typhil
4a	Resistant	12	12
4b	10	12	18
4c	Resistant	Resistant	Resistant
4d	12	15	15
4e	12	Resistant	10

Result & Discussion:
Chalcone(3a-e)were prepared by following the standard protocol[10] and were reacted with phenyl hydrazine to yield 3(furan-2-yl)1,5diphenyl 4-5-dihydro-1-H-pyrazole & its derivatives.the synthetic procedure for preparation of title compounds is given in scheme 1.The assigned structure & molecular formula of the newly synthesized compound (4a-e)were confirmed and supported by ^{1}H NMR as well as elemental analysis which was in full agreement with proposed structures.the compounds were screend in vitro antibacterial potential by disc diffusion method against pathogenic bacteria.The results of antibacterial activities expressed in terms of inhibition zone are reported in Table no.2.Even though the synthesized compound shows appreciable antibacterial activity.

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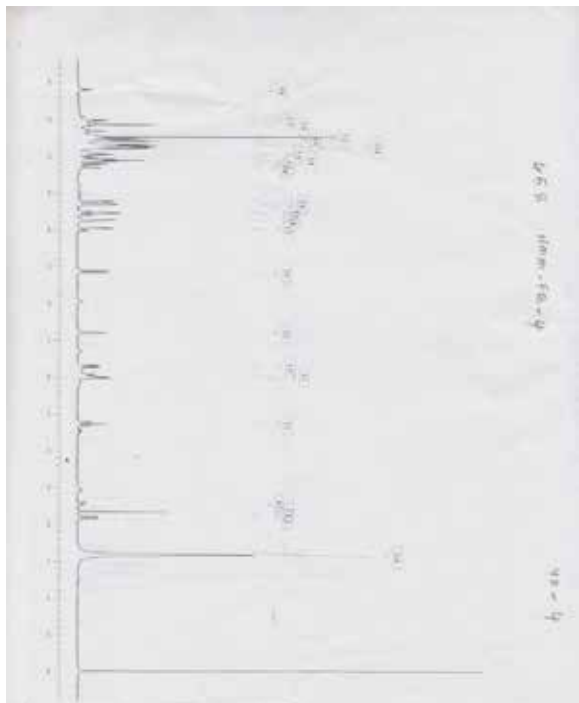
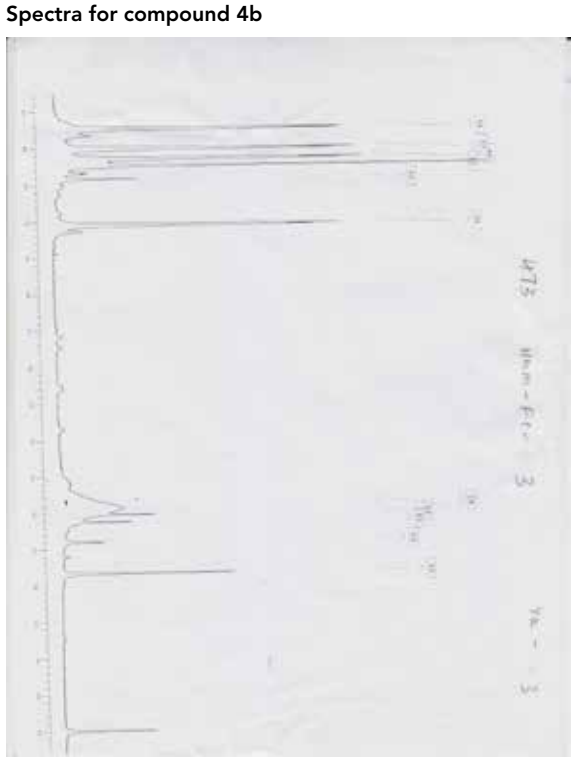
Spectra for compound 4a



Spectra for compound 4b



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