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SYNTHESIS OF BENZIMIDOLYL CHALCONES INTO N^1–SSUBSTITUTED PYRAZOLINES AND EVALUATION OF THEIR ANTIMICROBIAL ACTIVITIES.

NITIN SINGH SOLUNKEY* AND AMRENDRA KUMAR SINGH**

Declaration

The Declaration of the authors for publication of Research Paper in The Indian Journal of Research Anvikshiki ISSN 0973-9777 Bi-monthly International Journal of all Research: We, *Nitin Singh Solunkey and Amrendra Kumar Singh* the authors of the research paper entitled SYNTHESIS OF BENZIMIDOLYL CHALCONES INTO N^1–SSUBSTITUTED PYRAZOLINES AND EVALUATION OF THEIR ANTIMICROBIAL ACTIVITIES. declare that , We take the responsibility of the content and material of our paper as We ourself have written it and also have read the manuscript of our paper carefully. Also, We hereby give our consent to publish our paper in Anvikshiki journal , This research paper is our original work and no part of it or it's similar version is published or has been sent for publication anywhere else.We authorise the Editorial Board of the Journal to modify and edit the manuscript. We also give our consent to the Editor of Anvikshiki Journal to own the copyright of our research paper.

Abstract

1-Benzimidazolyl-3-aryl-prop-2-cne-1-ones (2) have been synthsised into N^1-substiluted pyrazoline derivatives by the interaction with phenyl hydrazine, thiosemicarbazide and hydraine hydrate in the presence of formic acid.The structure of newly synthesized compounds have been confirmed on the basis of their eiemntal analysis and spectral data. Newly synthesized compounds have been evaluated for their antimicrobial-activity in vitro against E. coil, P. aerugiiwsa and fungus P.oryzae, H.oryzae using paper disc method. Some of the compounds have shown significant activity against the pathogens.

Keywords : Benzimidolyl, pyrazolines, antimicrobial.

Introduction

The diverse nature of chemical reactions require various chemical strategies directed towards environmentally sound and ecofriendly methods and envisages minimum hazards as the performance criteria while designing the new process. One of the thrust areas for achieving this goal is to explore alternative reaction conditions to accomplish desired chemical transformation with elimination of the use of hazardous conventional solvents. Microwave irradia-tion (MWI) has gained popularity as a powerful non conventional tool for rapid, efficient and ecofriendly synthesis of a variety of compounds, because of selective absorption of microwave energy by polar molecules[1]. The application of MWI to provide enhanced reaction rate, improved yield and cleaner products[2] is proving quite successful in the

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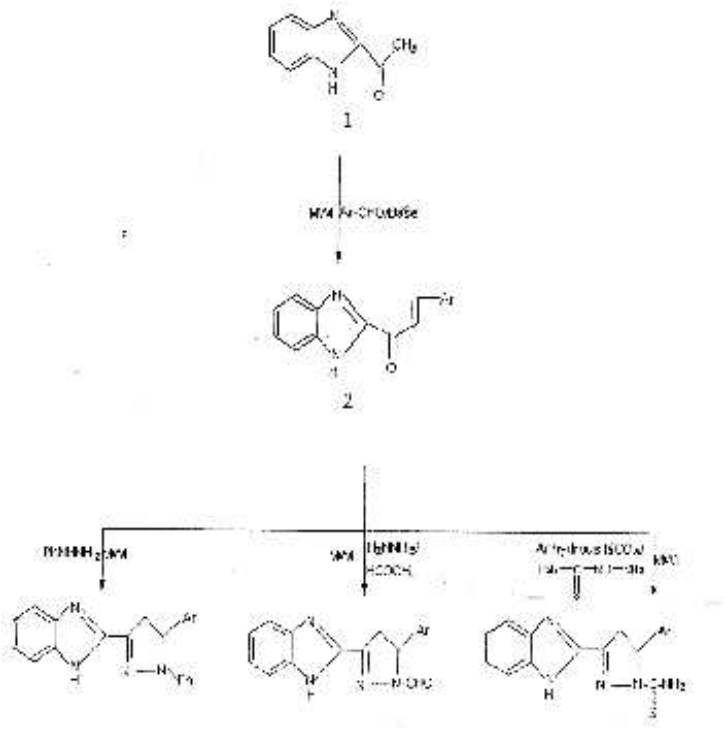
formation of a variety of carbon heteroatom bonds. The solvent free solid phase approach involving MWI exposure of neat reactants is applicable to rapid one pot assembly of heterocyclic compounds from in situ generated intermediate[3-5] Nitrogen-sulfur containing hetero cycle have been exclusively used as important pharmacophores and in drug designing. Bcnzimida zoles are important group of hcterocycles possessing a wide spectrum of pharmacological activities[6] such as antifungal[7], antihypertensive[8], antioxidant, cardio-vascular[10], anticonvulsant", antiviral[12], and H1V-IPR inhibitory activity[13]. Pyrazoline derivatives have been explored extensively due to their versatile biological activities. Antibacterial[15], antiamoebic[16]. Antiprolilerative[17], anticanccr[18], cox 11SS inhibitory[19] and cytoxic activities[20-23] have been reported to be associated with pyrazoline nucleus. Besides this, due lo easy accessibility and diverse chemical reactivity they are useful synthons. Therefore these two moieties were coupled and an attempt was made to synthesise molecules which may possess enhanced activities.

Result & Discussion

The required synthon l-benzimidazolyl-3-aryl-2-propen-1-one (2) was prepared by Claisen-Schmidt condensation of 2-acetyl benzimidazole (1) with substituted aromatic aldehydes in the presence of base under MWI by Literature method[24] .

Condensation of (2) with phenyl hydrazine gave N[1]-phenyl-3-benzimidazolyl-5-aryl-2-pyrazolines (3) in 75-80% yield. The reaction of (2) with hydrazine hydrate in the presence of formic acid afforded 3-benzimidazolyl-5-aryl-2-pyarzoline-l-carboxakldehyde (4) where as when (2) was condensed with thiosemicarbazide in the presence of anhydrous K_2CO_3, gave 3-benzimidazolyl-5-aryl-2-pyrazolinyl-l-thiocarbamides (5) (Scheme I). All these transformations were carried out using solvent less, solid phase neat reactants under microwave irradiation. The reaction was completed with in 4-8 min with 75-80% yield. Above transformation when carried out using conventional heating method under liquid phase, required 6-8 hr of healing and yields were 55-60% indicating the superiority of MWI method over conventional heating.



3 4 5

Scheme 1

The characterization of newly synthesized compounds was based on their elemental analysis (Table-1) and spectral data. IR spectra of newly synthesized compound gave absorption bands at 3420-3380 cm^{-1} (-NH of benzimidazole ring), 1440-1385 cm^{-1} (combined vibrations of O=N and N-N grouping), For compounds (4) a sharp peak at 1690 cm^{-1} for aldehydic group was obtained whereas for compounds (5) absorption band at 1360-1340 cm^{-1} for C=S linkage was observed.

^1H NMR spectra of these compounds gave signals at δ 3.36-3.38 (double doublet for C_4-A_A), δ 3.99-4.01 (double doublet C_4-H_B), and δ 6.18-6.22 (double doublet C_5-H_x), confirming the presence of typical ABX pattern

of pyrazolinc system. For compounds (4) a sharp high intensity singlet for aldehyde proton was observed at δ 10.01. Compounds (5) exhibited a sharp singlet at δ 6.75 for proton of amino group. (Table II).

The mass spectra of these compounds gave molecular ion peaks corresponding, to their molecular mass.

Experimental

The melting points reported were determined in open capillary and are uncorrected. The IR spectra were taken on a Perkin-Elmer spectrometer using- KBr pellets (v cm^{-1}). ^{1}H NMR were recorded on a Brucker DRX-300 spectrometer (Chemical shift δppm,$CDCl_3$) and mass spectra (FAB) were recorded on Jeol SX 102/DA 600 mass spectrometer using m-nitrobenzyl alcohol as matrix . The matrix peaks were erved at m/z 136, 137, 154, 289 and 307. The purity of products and progress of reaction was cheeked by TLC using ethyl acetate:benzene (1:9) as eluent and silica gel G as adsorbent. All the transformation were carried out in domestic microwave oven (Sam amg 1630N, output energy 800 watt, frequency 2450 MHz

T A B L E 1 *Physical dala of synthesized compounds*

T A B L E II *Spectral data of synthesized compounds*

Comp.	IR (KBr,v cm^{-1})	^{1}H NMR (CDCl$_3$ δppm	MS (FAB, m/z %)
3a	3022-2892 (CH str), 1590-1492 (C=N.N-N combined vib)	3.14-3.22(dd, 1H,C_4-H_A), 4.03-4.10 (dd, 1H,C_4-H_B) 5.59-5.65 (dd, 1H,C_S-H_X), 12.88 (s, 1H.NH bcnzimidazole)	338M+(75), 339M+1(100),227(4)
3b	3058-2837 (CH str), 1594-1498 (C=N.N-N combined vib)	3.35-3/17 (dd, 1H, C4-H_A). 3.97-4.07 (dd, 1H, C4-H_0), 5.31-5.37 (dd, 111, C5-I-I_X), 10.20 (8, 1H.NH bcnzimidazole)	368M+(80),369M-M(100), 261(50)
3e	2974-2890 (CH str), 1590-1450 (C=N.N-N combined vib)	3.33-3.41 (dd, 1H.C_4-H_A),4.01-4,11 {dd, 1H,C_4-I-I_B), 5.38-5.44(dd, 1M,C_5-H_X), 12.35 (s, 1H,NH bcnzimidazole)	372M+(85), 373M+1(100), 261(20)
5a	3425(NH), 3252(NH_2), 3057-2927 (CHstr), 2362 (OS) 1543-146K (C=N.N-N combined vib)	3.46-3.52 (dd, 1H, C,-H_A), 4,01-4.05 (dd, 1H, C.-H,,), 5.05-5.10 dd, 1H, C_sI-Ix), 7.028.20(m, 9H, Ar), 7,30 (s, 2H, NH_2), 12.3 (s, 1H, NH benzimidazole)	322M+1 (90), 321(60)
5c	3375 (Nil), 3275 (NH_2), 2932-2833 CM sir), 2364 (OS). 1599-1465 (C=N.N-N combined vib)	3.40-3.50 (dd, 11-1, C,-H_A), 3.84-3.99 (dd, ^{1}H, C_7 H) 5.99-6.02 (dd, 1II, C_5H_X), 7.78 (s, 2H, NH_2, 11.83 (s. 1H, NH bcnzimidazole)	382M+1(60),381M+(60), 289(40)
5d	M05 (Nil). 3252 (NH_2), 2933-28.10 (CU, 2360 (OS), 1599-I '165 (C=N.N-N combined vib)	3.40-3,52 (dd 11-1,C_4-1-I_A),3.92-4.0! (dd, 1H,C_4-M_B), 5.99-6.02, (dd. 1H, C_S-H_X), 7.! 1-7.71 (m, 6H, Ar), 7,30 (s. 211. NH_2), 12.37 (s, 1H, bcnzimidazole)	412M+1 (90),411M+(45), 378(40)
4a	3061-2960 (CH str), 1596-1495 (C=N.N-N combined vib)	3.29-3.36 (dd, 111, C_4-H_A), 3.83-3.93 (dd, 1H, C_4-H_B) (dd, 1H, C_5-H_X), 8.90 (s, 1H, CHO),	291M+l(100), 290M+(79) 237(25)
4c	l()-28'11 (CH str), 1595-1410 (C=N.N-N combined vib)	3.263.34(dd, IH, CH_A), 3.063.95(dd, 1H, C_4-H_a), 5.435,49 (dd, 1H,C_5H_X), 8.93 (s, 1M, CHO),	351M+1(100),350M+(70), 297(25)
4d-	2932-2893 (CH str). I596-M90 (C=N.N-N combined vib)	3.26-3.34 (dd, IH, C_4-M_A), 3.85-3.95 (dd, 1H. C_4-H_B), 5.44-5.49, Md, IH, C_5-H_X), 8.84 (s, 1H, CHO)	326M+1(100),325M+(80),237(25)
4f	2939-2875(CH str), 1510-1434 (C=N.N-N combined vib)	3.30-3.38(dd, 1H,C_4-H_A), 3.88-3.98 (dd, 1H, C_4-Hb), 5.39-5.45 (dd, IH, C_5-Hx), 8.80 (s, 1H, CHO)	34M+1(100),333M+(60)

General Procedure

MWI Process

Synthesis of 1-phenyl-3-benzimidazolyl-5-aryl-2-pyrazolines, 3a-f : Compound (2a-f; 0.01 mole) and phenyl hydrazine (0.012 mole) were mixed thoroughly to form an intimate mixture. It was then subjected to microwave irradiation at 300 watt power for 4-6 min. After completion of reaction as indicated by TLC, the reaction-mixture was cooled to RT and extracted with ethanol. The solid separated on standing was filtered and crystallized from benzene, petroleum ether to get analytical sample of 3a-f.

Synthesis of 3-bcnzimidazolyl-5-aryl-2-pyrazolinyl-1-carboxaldehydes, 4a-f : An intimate mixture of compound (2a-f; 0.01 moie) hydrazine hydrate (0.015 mole) and formic acid (15 mL) was subjected to MWI for 4-6 min at 240 wait power. After completion of reaction the separated solid was washed thoroughly with water, dried and crystallized from benzene-ethyl acetate to get analytical sample of 4a-f.

Synthesis of 1-Thiocarbamido-3-benzimidazolyl-5-aryl-pyrazolines, 5a-f : An intimate mixture of compound (2a-f; 0.01 mole), thiosemicarbazides (0.015 mole) and anhy-drous K_2CO_3 (4 g) was subjected to MWI at 300 watt for 3-5 min. After completion of reaction the reaction-mixture after

cooling at RT, was extracted with water and filtered. The residue obtained was washed again with water and dried. It was crystallized from ethanol to get analytical sample of 5a-f.

Synthesis of 1-phenyl-3-benzimidazolyl-5-aryl-2-pyrazoline, 3a-f : Compound (2a-f; 0.01 mole) and phenyl hydrazine (0.012 mole) were dissolved in absolute alcohol and reaction-mixture was refluxed for 6-8 hr on water bath. After cooling at RT the solid separated out was filtered, dried and crystallized to get analytical sample 3a-f.

T A B L E III *Biological screening results of compounds 3 amd 4 (Zone of inhibition in mm)*

Compd	E. coli	P. aeruginosa	P.oryzae	H.oryze
3b	10	12	11	12
3c	14	13	12	10
3d	12	11	10	10
3c	08	10	10	13
3f	12	16	14	14
4a	16	18	12	12
4b	13	16	10	10
4c	15	17	14	13
4d	14	19	12	10
4e	11	17	14	12
Ciproflaxange std.drug	20	23	20	20

Synthesis of 3-benzymidazolyl-5-aryl-2-pyrazolinyl-l-carboxaldehyde, 4a-f : Compound (2a-f; 0.01 mole) was taken in formic acid (50 mL). To it, hydrazine hydrate (0.015 mole) was added. The reaction-mixture was refluxed for about 6 hr and then left overnight at RT. The separated solid was filtered, washed with water and crystallized to get analyzed sample of 4a-f.

Synthesis of 1-thiocarbamido-3-benzimidazolyl-5-aryl-2-pyrazolines, 5a-f : Compound (2a-f; 0.0l mole) was dissolved in dry benzene (50 ml). To it, thiosemicarbazide (0.015 mole) and anhydrous. K_2 CO_3 (4 g) was added. The reaction-mixture was refluxed for about 7-8 hr. It was then left at RT. The solid separated was filtered, washed with little benzene, dried and crystallized to get analytical sample of 5a-f.

Anti Microbial Activity

The newly prepared pyrazoline derivatives were screened for their antimicrobial activity using paper disc method in vitro against E, coli, P. aemginosa and fungus P.oryzae, H.oryzae at a maximum concentration 250 ppm. The results were compared with standard drug ciproflaxange. All the compounds were found to possess moderate to good activity. (Table III)

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Refereces

[1.] Kappe C 0. Angew chem Int. Edn, 43, 6256 (2004).

[2.] Kidwai M & Molhsra P, Indian J Chem. 43B. 2330 (2006,).

[3.] Verma; R S, *"Microwave technology-chemical application"*, kirk T encyclopedia of chemical Technology, 5th Edn, (2004)

[4.]VERMA R S, *"Advances in green chemistry-chemical synthesis using microwave rradiation"* Astra-zeneca Research India Bangalore, (2002).

[5.]KAPPECO KUMAR D & VARRNA R S. Synthesis, 1795 (1999).

[6.]ENGUDRARD C. RENU, J.C. ALLOUCHI, H. LEGAR, M & GUIFFER H, Chem. Boll 48 938 (200).

[7.]INSUASTY B, FERNANDEZ F, QULRONG J, MARTINEZ R, GAVIN P &ANGCLCS, B, Ifeteracycl Comnum, 8151(2002).

[8.]SHAIFEE A, RASTKARI N & SHARIFZADCH, DARU, 12,81 (2004).

[9.]KHALRIL Z & YUANI SA, J Indian-Chem. Soc 58,168 (1981).

[10.]AURAM S. SAVED I, BOLOGO C & FLORIT M C, Cell Mol Med. 7(3), 287 (2003).

[11.]PANDEY V K, GUPTA V D & TIWARI D N, Indian, J Hetrocicl Chem, 14(4), 217 (2005).

[12.]AZARIFAR D & MASEUD S, Molecules, 7, 885 (2002).

[13.]ABID M & AZAM A, E.ur J Med Chem. 40, 935 (2005).

[14.]CHIMINCHI S, BOCCALINI M, HASSAN M M M, VIOLA G, DULL A F & CURINI M, Tetrahedron, 62, 90 (2006).

[15.]TANDON V K, YADAV D B, CHATURVEDI A K, & SHUKLA P K. Bioorg Med Chem Led, 15, , 3288 (2005).

[16.]KUMAR A. ARCHNA. SHARMA S. MALIK. V, SHARMA P, KAUSHIK K. SAXENA K. K, SRIVASTAVA-V K, VERMA R S, SHARMA 11, GUPIA S C & AGRAWAL, R C, Indian J Chem, 43B, 1532. (2004).

[17.]BHAT B A, DHAR K L, PURI S C, SAXENA A K, SHANMUGAVCL M & QUAZI G N, Bioorg Med Chem Lett. 15, 3177 (2005).

[18.]KRAZU J B & SYDNEY B, Chem Abstr, 72, 32043 (1970).

[19.]STADNYK L, CAMPBELL R S & JAHNSON B T, Bull. Environ Contain Toxicol, 6, 108 (1971).

[20.]SALLY ND, J Plmrmctcol, 90, 260 (1947).

[21.]NYATI M, RAO N S & SRIVASLAVA Y K, Asian J Chem, 757 (2008).

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