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Synthesis, Spectral Studies and Antimicrobial Activity of 3-(2'-n-butyl-4'-chloro-1-H- imidazol-5'-yl)-1-aryl prop-2-ene-1-ones

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Abstract: ***3-(2'-n-butyl-4'-chloro-1-H- imidazol-5'-yl)-1-aryl prop-2-ene-1-ones. (1a-1j) have been synthesized. The products have been assayed for their antimicrobial activity against Gram +ve bacteria and Gram –ve bacteria and fungi. The products have been characterised by IR, [1]HNMR, Mass Spectra and TLC.***
Keywords: Chalcones, antimicrobial assay

I. INTRODUCTION

Chalcones derivatives have been found to possess wide range of therapeutic activities as Anti-inflammatory [1-2], Antiallergic [3], Antitumor [4-5], Antispasmodic [6], Antiulcer [7-8], Anthelmintic [9-10], Anticancer [11-12], Antiviral and Anti-tubercular [13], Anti HIV [14], Bactericidal [15-16], Cardiovascular [17], Fungicidal [18-20], Insecticidal [21-23], Herbicidal [24] activity etc. 3-(2'-n-butyl-4'-chloro-1-H- imidazol-5'-yl)-1-aryl prop-2-ene-1-ones. **(1a-1j)** have been synthesized by condensation of 2-(n-butyl)-4-chloro-5-carboxaldo-1H-imidazole with aromatic aldehyde in the presence of aq. NaOH solution.

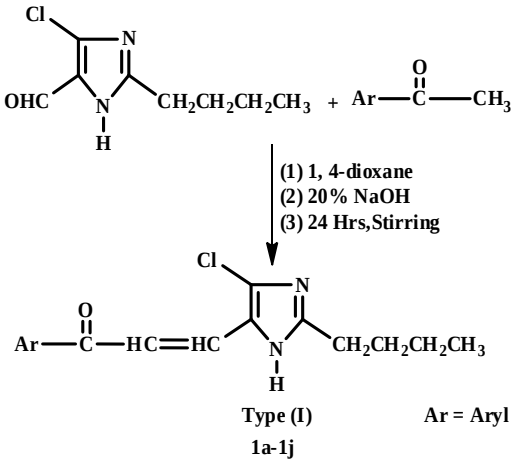
Chalcones have been proved to be an important intermediate for the synthesis of many heterocyclic compounds in organic chemistry. These facts prompted us to synthesize some new chalcone derivatives bearing substituted pyridine as a nucleus.

The products **(1a-1j)** were assigned by IR, [1]HNMR, mass spectral data, physical constants and antimicrobial activity are recorded in Table 1 and zone of inhibition that displayed by standard drugs are recorded in Table 2.

II. EXPERIMENTAL

All the melting points were measured by open glass capillary method and are uncorrected. IR absorption spectra (in cm^{-1}) were recorded on SHIMADZU-FT-IR-8400 spectrophotometer, frequency range: 4000-400cm^{-1} using KBr disc pallet method, ^{1}H NMR on 400 MHz Bruker Avance-III spectrometer using DMSO-d6 as a solvent and TMS as instrument standard and mass spectra on SHIMADZU-GC-MS QP-2010 Ultra. The purity of the compounds were routinely checked by TLC using silica gel-G.

III. REACTION SCHEME



Scheme-1 Synthesis of 3-(2'-n-butyl-4'-chloro-1-H-imidazol-5'- yl)-1-(4''-methoxy phenyl)-prop-2-ene-1-one.

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IV. ANTIMICROBIAL ACTIVITY

3-(2'-n-butyl-4'-chloro-1-H- imidazol-5'-yl)-1-aryl prop-2-ene-1-ones. **(1a-1j)** have been synthesized. The purified products were screened for their antibacterial activity. The antimicrobial activity was determined by cup plate method [25] at a concentration of 50 µg/ml using 1, 4-dioxane as a solvent. The activity was taken by Gram positive bacteria *B.megaterium, S. aureus,* Gram negative bacteria *Escherichia coli, and S. Taphimarium* and antifungal activity against *Aspergillus niger*. After 24 hrs. of incubation at 37°C, the zone of inhibition were measured in mm. The antibacterial activity was compared with the known standard drugs, viz, Ampicillin, Chloramphenicol, Norfloxacin and antifungal activity was compared with known standard drug viz. Fluconazole. The compounds (1a-1j) showing comparable antimicrobial activity and zone of inhibition that displayed by standard drugs are recorded in Table 2.

V. GENERAL PROCEDURE

A. Synthesis of 3-(2'-n-butyl-4'-chloro-1-H-imidazol-5'- yl)-1-(4''-methoxy phenyl)-prop-2-ene-1-one. (1i)

A mixture of 2-(n-butyl)-4-chloro-5-carboxaldo-1H-imidazole (1.87gm, 0.01M); 4-Methoxy acetophenone (1.50gm, 0.01M); 1, 4-dioxane (20ml); 20% NaOH (20ml) was stirred for 24 hrs. at room temperature. Completion of reaction was checked with TLC. The reaction mixture was poured into crushed ice, filtered it, dried it. The product was crystallised in 1, 4-dioxane. Yield: 77%; M.P.: 87°C; (Required: C: 64.05; H: 6.01; N: 8.79%; $C_{17}H_{19}ClN_2O_2$; Found: C: 64.05; H: 6.01; N: 8.70%).

IR (KBr): 2968 (C-H str. asym); 2864 (C-H str. sym); 1459 (C-H str. Def) 3060 (C-H str. aromatic); 1558 (C=C ring skeletal); 1166 (C-H i.p. (def)); 751 (C-H-str.def); 1600 (C-N str.); 1515 (C=N str.); 3415 (N-H str); 1600 (N-H bending); 1653 (C=O str.); 1459 (CH=CH); 728 (C-Cl); 1250 (C-O-C str.).

^{1}H NMR: 0.9 (T, 3H, -C<u>H$_3$</u>); 1.2-1.3 (m, 2H, -C<u>H$_2$</u>-CH$_3$); 1.5-1.6 (m, 2H, -C<u>H$_2$</u>-CH$_2$-CH$_3$); 2.6 (T, 2H, -C<u>H$_2$</u>-CH$_2$-CH$_2$-CH$_3$); 12.8 (S, 1H, -NH); 7.4 (d, 1H, -CH=C<u>H</u>-) 7.6 (d, 1H, -C<u>H</u>=CH-); 7.1 (d, 2H, Ar-H); 8.0 (d, 2H, Ar-H); 3.8 (S, 3H, -OC<u>H$_3$</u>).

m/z: 318, 283, 268, 253, 240, 225, 211, 200, 184, 167, 145, 135, 115, 107, 92, 77, 64, 43, 41, 40.

Similarly other compounds (1a-1j) were synthesized.

VI. TABLE

TABLE – 1: The Physical data and Antimicrobial activities of compounds (1a-1j)

Sr. No.	Ar	Molecular Formula	M.P. (°C)	Antibacterial activity Zone of Inhibition in mm.				Antifungal activity Zone of Inhibition in mm.	% Yield	% Nitrogen yield	
				Gram +ve bacteria		Gram –ve bacteria				Calcd.	Found
				B. mega.	*S. aureus*	*S. taphi.*	*E. coli.*	*A. niger*			
1a	C_6H_5-	$C_{16}H_{17}ClN_2O$	80	15	17	15	14	14	78	9.70	9.61
1b	3-OH.C_6H_4-	$C_{16}H_{17}ClN_2O_2$	100	12	12	17	16	17	81	9.19	9.12
1c	4-OH.C_6H_4-	$C_{16}H_{17}ClN_2O_2$	95	14	15	16	18	15	80	9.19	9.09
1d	3-NH_2.C_6H_4-	$C_{16}H_{18}ClN_3O$	120	18	17	19	15	16	86	13.83	13.76
1e	4-Cl.C_6H_4-	$C_{16}H_{16}Cl_2N_2O$	150	20	18	21	19	20	85	8.67	8.59
1f	4-Br.C_6H_4-	$C_{16}H_{16}BrClN_2O$	110	17	14	19	18	17	88	7.62	7.59
1g	3-NO_2.C_6H_4-	$C_{16}H_{16}ClN_3O_3$	97	14	21	15	16	21	79	12.59	12.30
1h	4-NO_2.C_6H_4-	$C_{16}H_{16}ClN_3O_3$	83	18	17	22	19	19	82	12.59	12.25
1i	4-OCH_3.C_6H_4-	$C_{17}H_{19}ClN_2O_2$	87	17	16	17	15	18	77	8.79	8.70
1j	3-NH_2, 2-OH.C_6H_3	$C_{16}H_{18}ClN_3O_2$	116	21	20	16	22	22	89	13.14	13.10

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Table 2. Compounds showing comparable antimicrobial activity with known Standard drugs. Zone of inhibition in mm.

	Ar	Antibacterial activity Zone of Inhibition in mm.				Antifungal activity Zone of inhibition in mm.
		Gram +ve bacteria		Gram –ve bacteria		
		B. mega.	*S. aureus*	*S. taphi.*	*E. coli.*	*A. niger*
1	Ampicillin (50µg/ml)	27	26	25	28	-
2	Chloramphenicol (50µg/ml)	29	28	27	25	-
3	Norfloxacin (50µg/ml)	32	30	24	27	-
4	Fluconazole (50µg/ml)	-	-	-	-	26

Compounds Code	Antibacterial activity Zone of Inhibition in mm.				Antifungal activity Zone of inhibition in mm.
	Gram +ve bacteria		Gram –ve bacteria		
	B. mega.	*S. aureus*	*S. taphi.*	*E. coli.*	*A. niger*
1a-1j	(1e) 4-Cl.C_6H_4- (20)	(1g) 3-NO_2.C_6H_4- (21)	(1e) 4-Cl.C_6H_4- (21)	(1j) 3-NH_2, 2-OH.C_6H_3- (22)	(1e) 4-Cl.C_6H_4- (20)
	(1j) 3-NH_2, 2-OH.C_6H_3- (21)	(1j) 3-NH_2, 2-OH.C_6H_3- (20)	(1h) 4-NO_2.C_6H_4- (22)		(1g) 3-NO_2.C_6H_4- (21)
					(1j) 3-NH_2, 2-OH.C_6H_3- (22)

VII. CONCLUSION

3-(2'-n-butyl-4'-chloro-1-H- imidazol-5'-yl)-1-aryl prop-2-ene-1-ones. **(1a-1j)** have been synthesized.

VIII. ACKNOWLEDGEMENT

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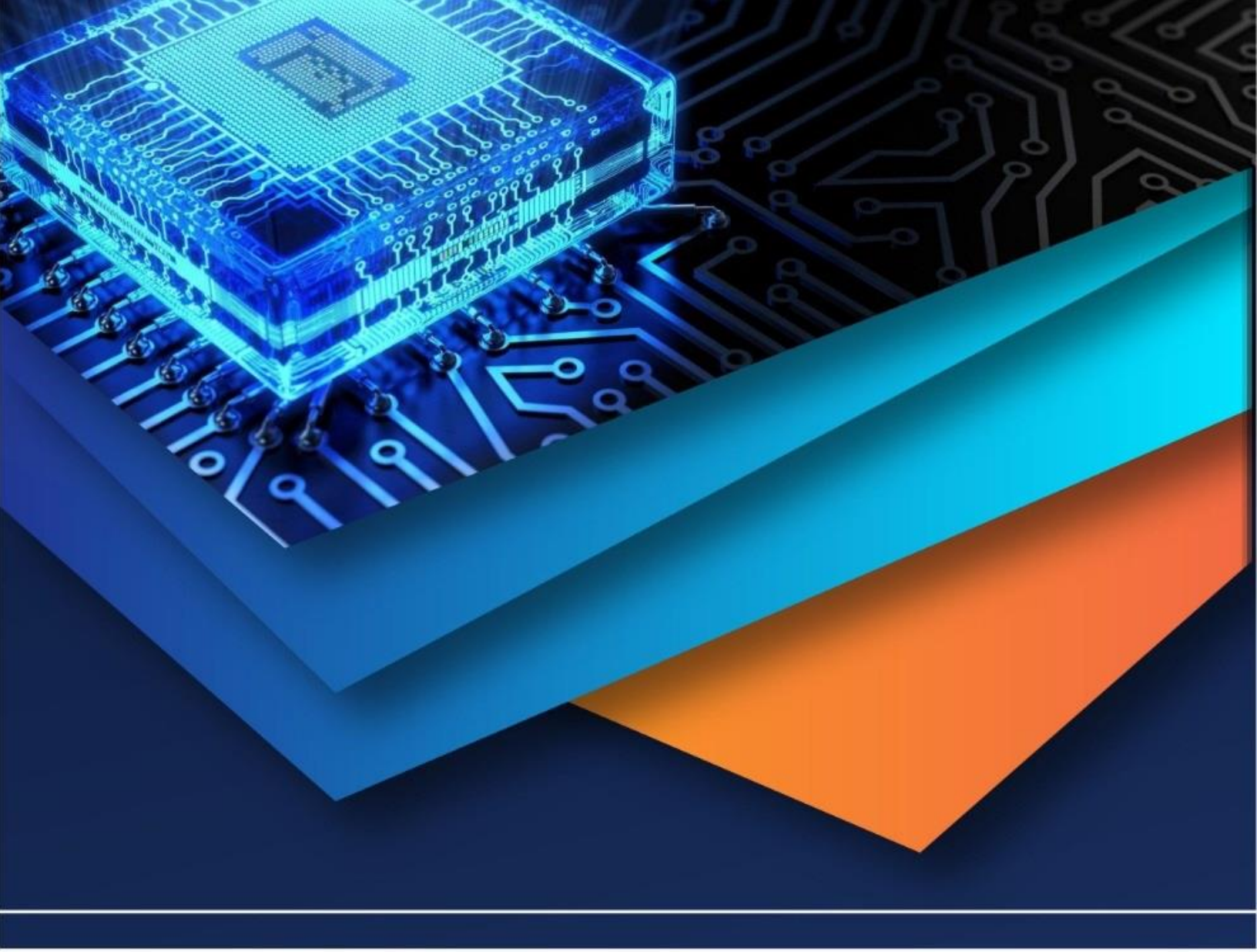

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