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Synthesis and characterization of 4-{4-(2-methyl-4-benzylidene-5-oxo-imidazol- 1-yl)phenyl}-6-(substitutedphenyl)-5,6-dihydropyrimidin-2-one and study of their antimicrobial actvities

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ABSTRACT

4-{4-(2-methyl-4-benzylidene-5-oxo-imidazol-1-yl)phenyl}-6-(substitutedphenyl)-5,6-dihydropyrimidin-2-one have been prepared by the refluxation for 3 hours of 4-benzylidene-1-{4-[3-(substitutedphenyl)prop-2-enoyl]phenyl}-2-methyl-imidazol-5-one with urea and potassium hydroxide in presence of ethanol . The intermidiate 4-benzylidene-1-{4-[3-(substitutedphenyl)prop-2-enoyl]phenyl}-2-methyl-imidazol-5-one synthesized by the condensation of 1-(4-acetylphenyl)-4-benzylidene-2-methyl-3,5-dihydro-imidazol-5-one with various aldehydes.

Key word : Synthesis, substituted chalcones, dihydropyrimidin.

INTRODUCTION

Literature survey reveals that most of the compounds having pyrimidine nucleus possess pharmacological action[1-3]. A wide spectrum of biological activities like anti-inflammatory,[4] antibacterial,[5] antifungal,[6] antitubercular,[7] analgesic and hypothermic[8] are found to be associated with compounds having pyrimidine moiety.

EXPERIMENTAL

Melting points were taken in open capillary tube and were uncorrected. IR spectra (KBr) were recorded on I.R. Spectrophotmeter of Buck scientific Model No. 500 and instrument used for NMR Spectroscopy was DUL 13C-1, 300 MHz and tetramethyl silane used as internal standard. Solvent used were $CDCl_3$ and DMSO. Purity of the compounds were checked by TLC on silica- G plates. Anti microbial activities were tested by Cup-Borer method.

Preparation of 4-benzylidene-2-methyl-1,3-oxazol-5-one (P-1).

In a 500 ml conical flask equipped with a reflux condenser a mixture of benzaldehyde (39.5g, 0.37M), acetyl glycine (29g, 0.25 M), acetic anhydride (63.5g, 0.62M) and anhydrous sodium acetate (15g, 0.183 M) was placed and heated on an electric hot plate with constant shaking. As soon as the mixture has liquefied completely, transfer the flask to a water bath and heat for 2 hours. Then add 100 ml of ethanol slowly to the contents of the flask, allow the mixture to stand overnight, filter the crystalline product with solution, wash with 25 ml of ice- cold alcohol and then finally wash with 25 ml of boiling water, dry at 100 ^{0}C. The yield of almost pure oxazolone was 76 % , m.p. 150 ^{0}C.Found: C(70.55%) H(4.82%) N(7.45%) , Calcd. for $C_{11}H_9NO_2$: C(70.58%) H(4.85%) N(7.48%).

Preparation of 1-(4-acetylphenyl)-4-benzylidene-2-methyl-imidazol-5-one (P-2)

In a 250 ml conical flask equipped with a

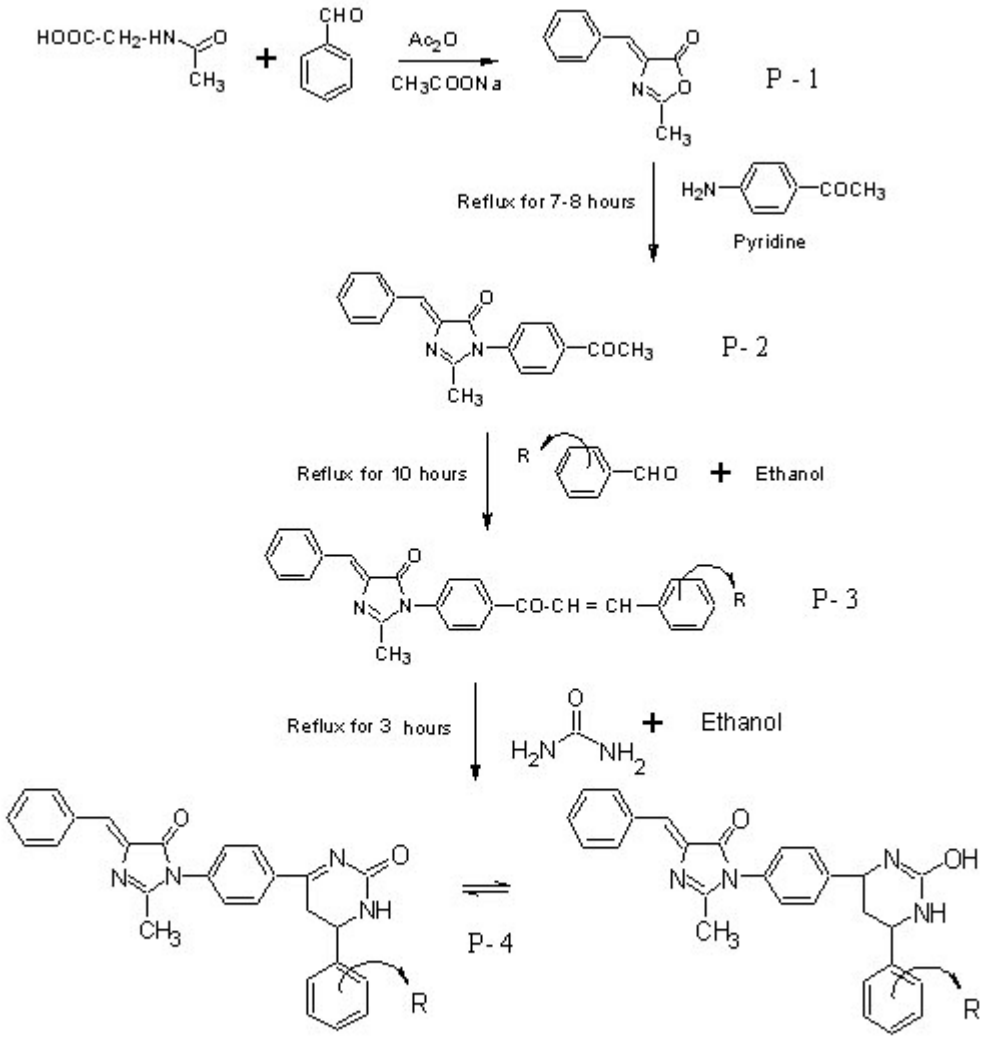
reflux condenser a mixture of 4-benzylidene-2-methyl-1,3-oxazol-5-one(18.719g, 0.1M), 1-(4-aminophenyl)ethanone (13.51g, 0.1M), 25 ml pyridine and about one pellet of KOH was placed and was heated on sand bath for 7-8 hours. Then the mixture was poured in ice. The precipitates were collected, washed with 10% HCl and re-crystallized from ethanol. The yield of the product was 72 % and the product melts at 112 ^{0}C.Found: C(74.94%) H(5.26%) N(9.18%) , Calcd. for $C_{19}H_{16}N_2O_2$: C(74.98%) H(5.30%) N(9.20%)

IR (KBr); (cm^{-1})

3080(= CH-), 2960(-CH Stretch), 1705 (>C=Oimidazolone),1650 (>C=N-), 1600(>C = C<),1375(-CH_3bend), 1260(C-N).

Preparation of 4-benzylidene-1-{4-[3-(substitutedphenyl) prop-2-enoyl]phenyl}-2-methyl-imidazol-5-one (P-3).

The solution of 1-(4-acetylphenyl)-4-benzylidene-2-methyl-imidazol-5-one(3.043g, 0.01M) in absolute ethanol (50 ml),substituted



4-{4-(2-methyl-4-benzylidene-5-oxo-imidazol-1-yl)phenyl}-6-(substitutedphenyl)-, 5,6-dihydropyrimidin-2-one

4-{4-(2-methyl-4-benzylidene-5-oxo-imidazol-1-yl)phenyl}-6-(substitutedphenyl)- 2-hydroxy-1,4,5,6- tetrahydropyrimidin

Scheme 1.

Table 1: Physical constant of 4-{4-[2-methyl-4-benzylidene-5-oxo-imidazol-1-yl]phenyl}-6-(substitutedphenyl)-5,6-dihydropyrimidin-2-one

No.	Sub.	R	Molecular Formula	Mol. Wt. (g/m)	Yield (%)	M. P. °C	Carbon (%)		Hydrogen (%)		Nitrogen (%)	
							Found	required	Found	required	Found	required
1	P-4a	-4-Cl	$C_{27}H_{21}ClN_4O_2$	468.93424	71	112	69.13	69.15	4.50	4.51	11.92	11.95
2	P-4b	-2-Cl	$C_{27}H_{21}ClN_4O_2$	468.93424	68	148	69.11	69.15	4.48	4.51	11.92	11.95
3	P-4c	-3-OCH_3,-4-OCH_3	$C_{29}H_{26}N_4O_4$	494.54114	65	132	70.41	70.43	5.28	5.30	11.30	11.33
4	P-4d	-2-NO2	$C_{27}H_{21}N_5O_4$	479.48674	66	230	67.61	67.63	4.40	4.41	14.60	14.61
5	P-4e	-2-OH	$C_{27}H_{22}N_4O_3$	450.48858	60	205	71.96	71.99	4.90	4.92	12.41	12.44
6	P-4f	-3-OCH_3,-4-OH	$C_{28}H_{24}N_4O_4$	480.51456	71	243	69.96	69.99	5.01	5.03	11.64	11.66
7	P-4g	-4-OH	$C_{27}H_{22}N_4O_3$	450.48858	64	202	71.96	71.99	4.91	4.92	12.42	12.44
8	P-4h	-4-$N(CH_3)_2$	$C_{29}H_{27}N_5O_2$	477.55698	69	276	72.92	72.94	5.68	5.70	14.63	14.66
9	P-4i	-4-OCH_3	$C_{28}H_{24}N_4O_3$	464.51516	63	202	72.38	72.40	5.20	5.21	12.03	12.06
10	P-4j	-3-OCH_3,-4-OCH_3,-5-CH_3	$C_{30}H_{28}N_4O_5$	524.56712	68	130	68.66	68.69	5.35	5.38	10.66	10.68

benzaldehyde (0.01M) and 2% NaOH (10 ml) were added and reflixed for 10 hours. After refluxing the reaction mixture was concen trated , cooled, filtered and neutralized with dil. HCl. The solid residue thus obtained was recrystallized with suitable solvent.

IR (KBr);P-3a (cm^{-1})

3080(= CH-), 2900 (-CH Stretch),1725 (>C=Oimidazolone) , 1675(>C=N-), 1590(>C = C<),1375 (-CH_3bend), 1260 (C-N), 700(C-Cl).

NMR ;P-3f

δ 2.501 , singlate (3H)(-CH_3), δ 3.490, singlate (3H)(-OCH_3), δ 5.631, singlate (1H) (=CH-vinylic),δ 6.660-7.902 , multiplate (14H) (Ar-H) 8.262, singlate(1H) (-OH) .

Preparation of 4-{4-[2-methyl-4-benzylidene -5-oxo-imidazol-1-yl]phenyl}-6-(substituted phenyl)-5,6-dihydropyrimidin-2-one (P-4)

A mixture of 4-benzylidene-1-{4-[3-(substitutedphenyl)prop-2-enoyl]phenyl}-2-methyl-imidazol-5-one(0.01M) ,urea(0.01M) and 1g. of potassium hydroxide(KOH) in 30ml of ethanol was refluxed for 3 hours. After standing overnight the solid formed was collected and crystallised from acetone.

δ

IR (KBr);P-4g (cm^{-1})

3350(>NH),3240 (-OH), 3090(= CH-) , 2950(-CH Stretch), 1720 (>C=O) ,1600 (>C=N-), 1160 (C-N), 1500(>C = C<),1460(-CH_2-bend),1400 (-CH_3 bend), 1240 (C-O),

NMR ;P-4e

δ 1.123, doublet (2H)(-CH_2-), 2.258, triplet (1H)(-CH<), 2.490,singlate (3H)(-CH_3), 3.361, singlate (1H)(-NH-), δ 5.416, singlate (1H) (=CH-vinylic), δ 6.588-7.944, multiplate (13H) (Ar-H), δ 8.097, singlate (1H)(-OH).

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Table 2: Antimicrobial activities of 4-{4-[2-methyl-4-benzylidene-5-oxo -imidazol-1-yl]phenyl}-6-(substitutedphenyl)-5,6-dihydropyrimidin-2-one

S. No.	Comp. No.	R	Zone of inhibitions in mm		
			E.coli	S.aureus	C.albicans
1	P-4a	- 4-Cl	19	20	22
2	P-4b	- 2-Cl	17	18	19
3	P-4c	- 3-OCH_3, -4-OCH_3	18	17	18
4	P-4d	- 2-NO_2	15	15	NA
5	P-4e	- 2-OH	15	15	17
6	P-4f	- 3-OCH_3, -4-OH	16	14	19
7	P-4g	- 4-OH	15	15	14
8	P-4h	- 4-$N(CH_3)_2$	15	16	18
9	P-4i	- 4-OCH_3	15	NA	17
10	P-4j	- 3-OCH_3, -4-OCH_3, -5-OCH_3	18	17	20
11	Penicillin	-	18	20	-
12	Kanamycine	-	19	24	-
13	Baycor 25 w.p.	-	-	-	24
14	Amphotericine	-	-	-	21

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